

The Journal *of the* SOCIETY OF DYERS AND COLOURISTS

Volume 68 Number 10

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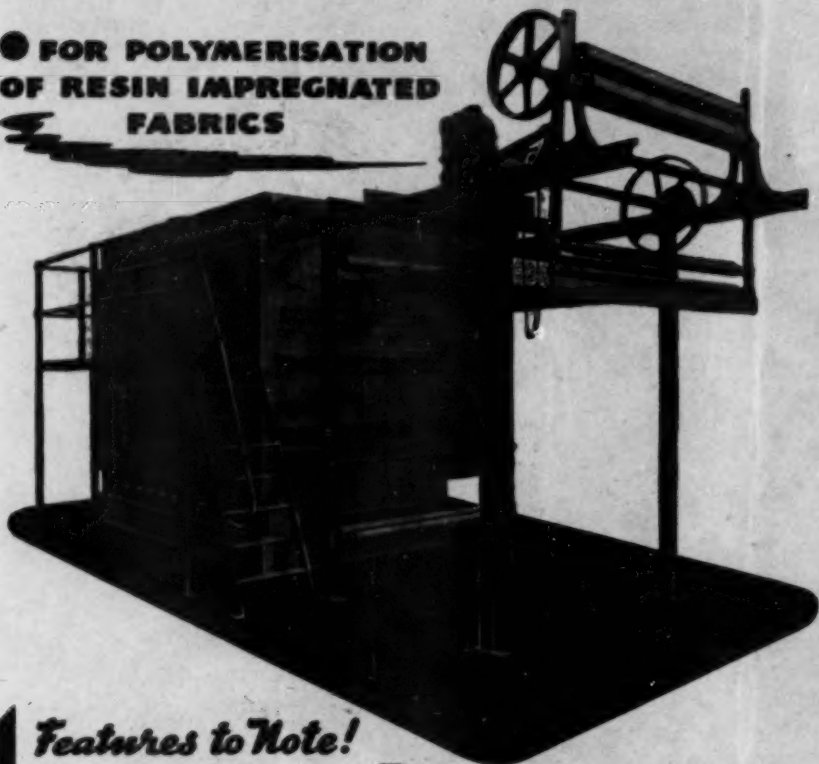
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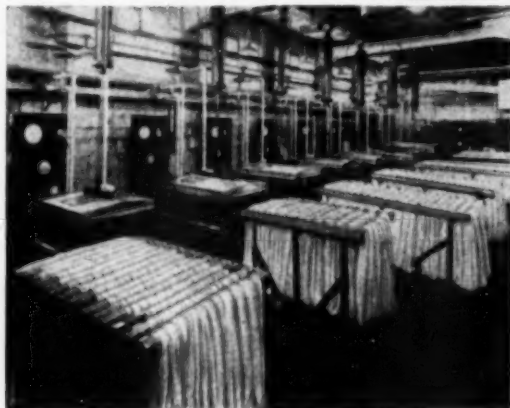
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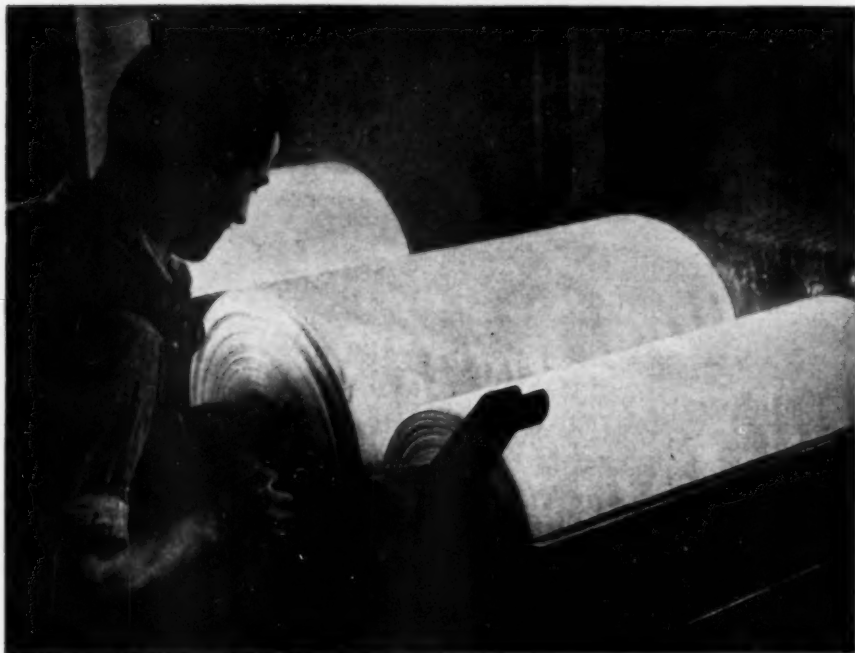
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Volume 2

Compiled and published jointly by the Textile Institute
and the Society of Dyers and Colourists

W J HALL
A.R.C.S. B.Sc.

Editors

C J W HOOPER
Ph.D. D.I.C. A.R.C.S.

The Textile Institute and the Society of Dyers and Colourists have jointly published a Review of Textile Progress, Volume 2 relating particularly to the year 1950. There are 25 contributors in the Review and the authors are experts in the various sections for which they are responsible

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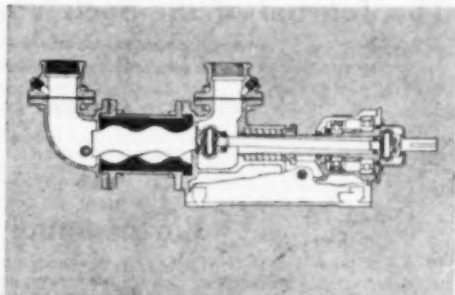
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NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring general information regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1-6 of the January 1952 and pages 237-240 of the July 1952 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, 19 Piccadilly, Bradford, Yorkshire (Telephone Bradford 25138-9). Editorial Communications should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

LECTURES

Colour Photography

R. B. Collins and C. H. Giles

Turkey Red Dyeing in Scotland—its Heyday and Decline

R. A. Peel

Non-felting Wool and Wool Mixtures (Mercer Lecture)

F. C. Wood

COMMUNICATION

Combination of Wool with Acids

B. Olofsson

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Forthcoming Meetings of the Society (see also page xxvii)

MANCHESTER SECTION

Unless otherwise stated meetings are held in the Textile Institute, 10 Blackfriars Street, Manchester 3, and commence at 6.30 p.m.

1952
Thursday 23rd Oct. Dr. R. L. Wormell. *Researches on Regenerated Protein Fibres* (Joint meeting with the Textile Institute, Lancashire Section)

Wednesday 12th Nov. L. Higgins, Esq., M.Sc. (I.C.I. Ltd.). *Dye-stuffs Div.*. *The Dyeing of Effect Threads*. Nicolson Institute, Leek

Friday 21st Nov. LADIES' EVENING. Manchester College of Technology, commencing at 7 p.m. *Penny Plain. Twopence Coloured*. Lectures and demonstrations on the miscellaneous applications of dyestuffs. Speakers to be announced later

Friday 19th Dec. Lecture to be announced later. Details to be announced later

1953
Friday 16th Jan. G. G. Taylor, Esq. (Clayton Aniline Co. Ltd.). *The Measurement of Colour*

Friday 20th Feb. W. Speke, Esq., B.Sc. (I.C.I. Ltd., Dye-stuffs Div.). *High Temperature Dyeing of Viscose Rayon*

Friday 13th March Manchester College of Technology. Afternoon and Evening Symposium on *New Fibres*. Details to be announced later

Friday 20th March Joint meeting with the British Association of Managers of Textile Works. Midland Hotel, Manchester. Lecture by Dr. Fargher (Shirley Institute). Details to be announced later

Friday 17th April ANNUAL GENERAL MEETING. Details of lecture to be announced later

NORTHERN/IRELAND SECTION

All Meetings to be held in Queen's Hotel, Belfast at 7.30 p.m.

1952
Wednesday 12th Nov. J. C. Brown, Esq. and G. G. Taylor, Esq. (Clayton Aniline Co. Ltd.). *Microscopy for the Dyer and Finisher*

Wednesday 10th Dec. A. S. Cluley, Esq. (Courtaulds Ltd.). *Some Observations on Practice in the Dyeing and Finishing of Rayon Materials* (Joint meeting with Textile Institute)

1953
Wednesday 14th Jan. E. R. Wiltshire, Esq. (I.C.I. Ltd.). *The Control of Azoic Dyeing Processes* (Joint meeting with Foremen Dyers Guild)

Wednesday 11th Feb. F. H. Marsh, Esq. (Longclose Engineering Co. Ltd.). *Pressure Dyeing and Bleaching with Possible Indication of New Developments*

Wednesday 11th March Dr. B. C. Gee. *The Educational Side of Textile Processing*

March/April ANNUAL GENERAL MEETING and DINNER (date will be announced later)

LONDON SECTION

All meetings held at 6 p.m. in the Rooms of the Royal Society, Burlington House, Piccadilly, London W.1, unless otherwise stated

1952
Friday 7th Nov. J. A. Radley, Esq., M.Sc. (Lond.), F.R.I.C. *Fluorescent Effect* (there may be a slight amendment to this title)

Friday 5th Dec. Dr. P. R. Peacock (Royal Beatson Memorial Hospital, Glasgow). *Food Dyes*

1953
Friday 9th Jan. *Uses of Microscopy in Textile Dyeing and Finishing* (Clayton Aniline Co. Ltd.)

Friday 6th Feb. F. Ward, Esq. (Courtaulds Ltd.). *Methods and Principles of Textile Fireproofing*

Friday 6th March Title to be announced later

MIDLANDS SECTION

Except when otherwise stated, Lecture Meetings commence at 7 p.m.

1952
Wednesday 29th Oct. G. H. Lister, Esq., Ph.D., B.Sc. *Some New Angles on Detergency*. College of Technology, Leicester

Wednesday 19th Nov. A. E. Stubbs, Esq., Ph.D., F.R.I.C. *Developments in Cotton Bleaching at home and abroad*. Victoria Station Hotel, Nottingham

Saturday 22nd Nov. LADIES' EVENING—DINNER DANCE. Black Boy Hotel, Nottingham

Wednesday 17th Dec. *Colloquium*. Short papers on practical problems given by Local Members. King's Head Hotel, Loughborough

1953
Wednesday 28th Jan. J. Boulton, Esq., M.Sc. (Tech.), F.R.I.C., F.T.I. *Modern Dyeing Theory for the Plain Man*. Masonic Hall, Hinckley, 7.30 p.m. (Joint meeting with the Textile Institute and Hinckley Textile Society)

Thursday 19th Feb. E. M. Walker, Esq., F.T.I., F.R.M.S. *The Chemist and Technologist in the Hosiery and Knitted Goods Industry*. (Joint meeting with the Textile Institute). Carpet Trades Canteen, Kidderminster, 7.30 p.m.

Wednesday 25th Feb. E. R. Wiltshire, Esq., B.Sc. *Azoic and other Fast Dyes on Knitted Cotton Fabric*. College of Technology, Leicester

Friday 13th March MIDLANDS SECTION DINNER. Royal Hotel, Leicester

Wednesday 18th March G. H. Osborn, Esq., F.R.I.C., A.M.Inst. M.M. *Colour in Chemistry* (Joint meeting with the British Association of Chemists). School of Arts and Crafts, Derby

Wednesday 22nd April ANNUAL GENERAL MEETING OF THE SECTION. Gas Board Theatre, Nottingham

Towards the end of the session Dr. H. White (Textile Research Institute, Princeton, U.S.A.) will lecture in Nottingham. Particulars later

Continued on page xxvii

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Volume 68—Number 10

OCTOBER 1952

Issued Monthly

Proceedings of the Society

A New Method of pH Control in Dyeing and Some Observations on the Metachrome Process

R. J. HANNAY, W. H. MAJOR, and R. PICKIN

Meeting of the West Riding Section held at the Victoria Hotel, Bradford, on 13th March 1952, Mr. C. O. Clark in the chair

The rise in the pH of the dye liquor which normally occurs in metachrome dyeing, particularly in closed machines, can be prevented or even reversed, simply by dissolving an ester such as diethyl tartrate or ethyl lactate in the bath at the commencement of dyeing. These esters, under the dyebath conditions, hydrolyse to produce free acid at a suitable rate. Strength tests showed that wool suffered no more damage when dyed by the metachrome process, with or without the ester, than when dyed with acid dyes. The use of these esters in metachrome dyeing, and in dyeing with acid milling and Carbolan (ICI) dyes, gives more level results than conventional methods, with no adverse effect on the exhaustion of the dyebath.

The metachrome process of dyeing has been the source of considerable discussion during the past four or five years, and opinions as regards its merits and possible drawbacks show some conflict. Statements have appeared in the technical press from time to time asserting that this method of dyeing chrome dyes on wool gives rise to more than normal degradation of the wool, and it has been stated that loose wool and slubbing dyed by this method have poorer spinning qualities than when dyed afterchrome. The authors' experience has been that such criticisms have always come from dyers of loose wool, slubbing, or yarn, and no complaint from piece dyers has been received. The main basis of such criticism rests largely on the pH value of the metachrome dyebath, which is on the alkaline side, and tends to rise during the dyeing process, particularly when dyeing in a completely closed system. The published evidence that any damage to wool occurring during metachrome dyeing is due to that process is by no means conclusive, and it is known that many dyers have successfully applied this process without trouble. It was, therefore, felt that a fuller investigation of the reactions which occur during the dyeing of wool by this process might yield some useful and practical results.

A brief review of some of the work already published on the dyeing of chrome dyes on wool will indicate the position which led to the work described in the present paper. Stevens, Rowe, and Speakman¹ examined the metachrome dyeing process, and showed that it depended on the simul-

taneous dyeing and mordanting of the wool from a slightly alkaline dyebath, the chromium mordant being taken up from the dyebath as sexivalent chromate ions, in which form no lake of the dye is produced. Hydrolysis of the sodium chromate is accompanied by the formation of an equivalent amount of caustic soda. The ammonium sulphate present in the bath reacts with this alkali to give sodium sulphate and ammonia, and provided this ammonia can escape the pH of the bath cannot rise. During the course of the dyeing the wool reduces the sexivalent chromate ions to trivalent chromic ions, which then form the dye lake. In practice the net result is a fall of something like 0.5 in the pH value of the bath in a boiling time of 1½–2 hr. This fall is increased if boiling is continued longer. This work was followed up by Carlene, Rowe, and Speakman², who made a careful study of the chroming of wool and again showed that the presence of ammonium salts in the bath had a restraining effect on any rise in pH during the process. It must be stressed that the results recorded in these two papers were all obtained in open-bath systems, where any ammonia evolved was free to escape from the system. It is of interest to note in addition that in the first paper the authors draw attention to some earlier work, by Speakman and Coke³ and by Liu, Speakman, and King⁴, and restate—

The metachrome process not only saves time and labour, but the conditions of dyeing are such that the wool is damaged to a smaller extent than in other methods of dyeing with chrome mordant dyes.

This contention is largely supported by Carter² where he states—

From the results in the above table it is possible to obtain some idea of the relative merits of the different dyeing techniques. The following list is in order of merit, starting from the one which has the least degrading effect on the wool—

- (a) Afterchrome on acetic acid dyeing
- (b) Metachrome mordant method
- (c) Neutral dyeing
- (d) Acid dye with acetic acid
- (e) Acid dye with formic acid
- (f) Acid dye with sulphuric acid
- (g) Afterchrome on sulphuric acid dyeing.

In 1948 Peryman⁶ and Sutcliffe⁷ drew attention to the fact that in commercial practice the rise in pH during dyeing by the metachrome process was not always prevented, and gave examples of a considerable rise during dyeing, amounting in some cases to as much as 1.4 pH units and resulting in a final dyebath pH in the region of pH 9. Gaunt⁸ followed this up and showed that the rise in pH was governed to a large extent by the type of machine which was used for dyeing. He showed that with an open machine the pH fell, as stated by the earlier workers, but that in the case of closed machines the pH rose in accordance with the efficiency with which the machine prevented the escape of ammonia from the system. It has been stated that treating wool at the boil in baths of pH 8.5–9.0 is sufficient to cause some damage to the wool, but Gaunt is careful to emphasise that it has never been conclusively proved that fibre damage does occur when dyeing wool by the metachrome process. It must be admitted, however, that at first sight there is an apparent risk in treating wool in baths of such alkalinity, and if no products were present in the dyebath other than the alkali, damage would be fairly certain. The effects of the chromium salts, ammonium salts, and dye present must, however, be taken into consideration. Some work has already been done at Leeds University on the more fundamental aspects, and Corker⁹ and Thornton¹⁰, working with carefully buffered solutions starting at pH 7 and stepping up to pH 9.5, have shown that damage to the wool increases progressively with rise in pH and with length of boiling time. The action of the boiling buffer solutions on the wool was the same in the presence of dye. The addition of potassium chromate to the solutions, however, appreciably reduced the damage to the wool, whilst an intermediate degree of damage was observed when both potassium chromate and dye were added. It must be emphasised here that the whole of this work was carried out in the absence of ammonium salts, which are normally present in a metachrome dyebath, and which, the present authors feel, would considerably modify the results obtained. It being realised that there is an increasing use of totally enclosed circulating dyeing machines in the industry, the use of which might give rise to difficulties, an investigation of the metachrome process under these enclosed conditions was undertaken.

A laboratory apparatus was designed, and was fitted with a reflux condenser to ensure that there

was no escape of ammonia from the system. The use of this apparatus confirmed that the pH of the metachrome dyebath rises appreciably in a closed system, and that the rise is steady throughout the period of dyeing. The maximum final pH that was found in any experiment was 9.2, and that was reached in only one case, when an addition of ammonia had been made to the dyebath at the beginning in order to aid the dissolution of the dye, a procedure not uncommon in works practice. Generally speaking, the final pH values were between 8.6 and 9.0.

A method was then sought of preventing this rise in pH during dyeing in such a way as not to affect the dyeing system by causing unevenness, lack of penetration, or impairment of fastness properties. Two important points have to be borne in mind when seeking a solution to this problem—

(1) Whatever addition is made to the dyebath, the substance added must be compatible with the dye and the mordant present and have no deleterious effect on the wool.

(2) The initial pH of the normal metachrome dyebath should not be materially reduced, otherwise there is considerable risk of precipitating some of the metachrome dye and so producing uneven dyeings. This condition precludes the direct addition of acid or acidic salts to the dyebath at the commencement of dyeing, and it is not easy to make controlled additions of such substances during the course of dyeing in a manner conducive to uniform results.

Other people have been working on this problem, and Gaunt⁸ mentioned a suggestion by Lister that the replacement of ammonium sulphate by a good buffer such as ammonium dihydrogen phosphate might prove to be a useful modification of the process. As will be shown later (p. 376) it is believed that such a buffer could not be so beneficial to the process as is ammonium sulphate. The writers' experience and the evidence of previous workers show that ammonium sulphate plays a reliable and useful part in the metachrome process. It is cheap and readily available, and its use should be continued.

Consideration was given to the possibility of making an addition to the dyebath of a neutral or nearly neutral product which during the course of dyeing would liberate an acid at approximately the same rate as alkali is produced by the hydrolysis and reduction of the chrome mordant, and attention was turned to an examination of hydrolysable organic esters and organic derivatives of acids. There is a vast number of such products, the majority of which fall into two main classes—

- (a) Those which are relatively stable and difficult to hydrolyse
- (b) Those which are easily hydrolysed.

Between these groups there is a comparatively small range of products which hydrolyse at a rate suitable for this purpose. In the choice of an ideal product it was considered that three further conditions should be imposed, although failure to comply with

any one of them does not mean that a product will not give the desired result—

- (i) The products of hydrolysis should be an acid and a substance as nearly neutral as possible rather than an acid and a weak base
- (ii) The original product should hydrolyse in slightly alkaline solution
- (iii) The acid produced by the hydrolysis should be, if possible, non-volatile in the dyebath.

One product which immediately came to mind, because it was already established in another section of the textile industry, was diethyl tartrate, whilst a second was gluconic lactone. Experiments were therefore put in hand to study the effect of small additions of these products to the metachrome dyebath, using the totally enclosed apparatus mentioned earlier. It was anticipated that the ester would hydrolyse at the boil to give free tartaric acid and that the lactone would hydrolyse to give gluconic acid, the free acids then being available to react with any alkali or ammonia formed during dyeing.

These ideas were tested by measuring the pH of the dye liquors during the dyeing process. It was not practicable to measure directly the pH of the boiling dyebath, and a continuous sample was withdrawn by a metering pump, cooled to room temperature, passed through a vessel containing the pH electrodes and temperature compensator, and returned cold to the dyebath. In this way a standard liquor ratio and a steady boil were maintained throughout the dyeings. The rate of sampling was 25 ml./min., the dyebath contained approx. 600 ml. of liquor, and the sampling and measuring system held approx. 75 ml.

It was recognised that, while this procedure overcame the practical difficulty of measuring the pH of the boiling dyebath, it introduced some uncertainties, for it did not necessarily follow that the pH of the cooled solution was the same as that of the boiling liquor. This point was emphasised by the fact that the metachrome mordant solution undergoes a reversible colour change when it is heated and cooled, indicating that the ions present in the hot and cold solutions are different.

A series of experiments was therefore made to investigate the change with temperature of the pH of the following solutions—

- (1) 0.05 M. Potassium hydrogen phthalate
- (2) 0.05 M. Sodium tetraborate (borax)
- (3) McIlvaine phosphate buffer solution of nominal pH 8.0
- (4) A metachrome dye liquor of the following composition—

Metachrome Brilliant Blue SRL ... 1.0 g.
Metachrome mordant ... 1.0 g.
1.5 N. Ammonium hydroxide ... 1.0 ml.
Distilled water, to make ... 600 ml.

- (5) A dye solution as (4) which had been used to dye a 20-g. hank of scoured wool
- (6) A dye solution as (4) to which had been added 1.5 g. diethyl tartrate, and which had been used to dye a 20-g. hank of scoured wool.

In preparing liquor (5) the wool was entered into a liquor of composition (4) at 50°C. in the closed apparatus already described, and the temperature was raised to the boil during 45 min. and maintained at the boil for 90 min. The wool was withdrawn, and the exhausted liquor cooled and filtered. Solution (6) was obtained similarly except that the diethyl tartrate was added to the bath immediately before the wool was entered.

It will be seen that these six solutions comprise three recognised buffer solutions, a normal metachrome dye liquor, the same metachrome dye liquor exhausted in the normal manner, and the metachrome dye liquor exhausted after the addition of diethyl tartrate.

Samples of each solution were placed in a thermostat at temperatures ranging from 15° to 65°C., and the e.m.f. given by a calomel electrode-glass electrode system in each solution was measured at each temperature. Then by assuming the pH values of the potassium hydrogen phthalate solution¹¹ at each temperature, the pH of the other solutions at that temperature could be calculated. These pH values are plotted in Fig. 1, which also includes a plot of the change in the pH of the neutral point with temperature. The latter pH values are equal to 0.5 p*K_w*, where *K_w* is the ionisation constant of water at each temperature¹².

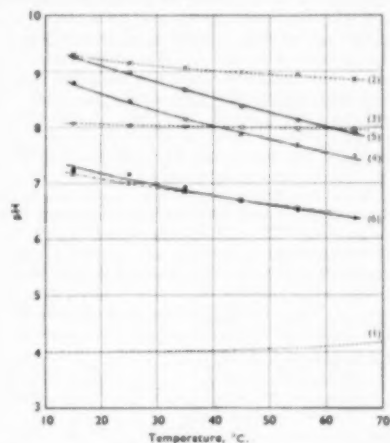


FIG. 1

It will be seen that the curves for the metachrome dye liquors (4-6) are reasonably parallel over this temperature range, and it has been assumed that a similar relation holds up to the boiling point. If this is so, then since changes in the pH of these dye liquors with temperature are similar, although the

pH of the cooled continuous sample of the dyeing experiments does not give the pH of the boiling liquor, any changes in the pH of the boiling solution will be fairly well reproduced in the pH of the cooled sample. The pH measurements throughout the subsequent work were made at about 15°C., and the above relation between pH and temperature must be borne in mind in assessing them.

A further point of interest to be noted from Fig. 1 is that the slopes of the metachrome dye liquor curves are very similar to that of the neutral point curve. This is in marked contrast to the curves for the alkaline buffer solutions (2, 3), which are almost parallel to the temperature axis, and therefore diverge from the neutral point curve. Thus as the temperature of these buffer solutions is raised their pH remains fairly constant, but in relation to the neutral point the solutions become progressively more alkaline. This suggests that, if the mordant solution were strongly buffered, so as to minimise the rise in pH inherent in the dyeing mechanism, the alkalinity at the boil would be higher than that given by the standard metachrome mordant solution and one would expect the wool to suffer greater damage.

Having established that the pH readings made on the cold circulating sample of dye liquor were closely related to the conditions in the dyebath, a number of dyeing experiments were made. In each case a 20-g. hank of 2/23/11 undyed worsted yarn and 600 ml. of liquor were used, thus giving a 30:1 liquor:wool ratio. The cold liquor was allowed to circulate through the sampling system for 15 min. before the hydrolysing material was added, and the dyebath was then heated with a constant electrical input so that approx. 45 min. were taken for the liquor to reach the boil from 50°C.; the boil was maintained for a further 90 min. The wool was entered into the liquor when the temperature reached 50°C. and was agitated from time to time. The pH of the cooled sample was read at intervals of 5 min., the resulting curves being plotted in Fig. 2. In this graph zero time is the time at which the wool was entered into the liquor, i.e. when the liquor temperature was 50°C.

The following dyebath recipes were used—

(i)	Metachrome Brown B	1.0 g.
	Metachrome mordant	1.0 g.
	Ammonia (5% soln.)	0.5 ml.
	Distilled water, to make	600 ml.
(ii)	As (i) together with—			
	Diethyl tartrate (500 g./litre alcoholic soln.)	1.0 ml.
(iii)	As (i) together with—			
	Gluconic lactone	0.86 g.
(iv)	As (i) together with—			
	Ammonium gluconate	1.0 g.
(v)	As (i) but omitting the ammonia			
(vi)	As (ii) but omitting the ammonia			
(vii)	As (i)			
(viii)	As (ii)			
(ix)	As (iii)			

The weights of gluconic lactone and ammonium gluconate in (iii) and (iv) are equivalent to the tartaric acid in the diethyl tartrate of (ii).

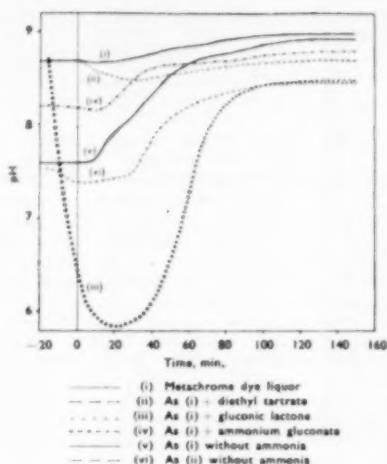


FIG. 2

Curve (i) shows how, in a typical metachrome dyeing, the pH increases as dyeing proceeds, and curve (ii) shows how the addition of diethyl tartrate prevents this rise. Gluconic lactone (iii) hydrolyses too rapidly at the beginning of dyeing, and ammonium gluconate (iv) depresses the initial pH of the dyebath, both features being undesirable.

It will be seen on comparing curves (i) with (v), and (ii) with (vi), that omitting the ammonia from the dyeing recipes decreases the initial pH of the liquor by about 1.1, but, in spite of this, the final pH of the liquor is only 0.1–0.2 lower.

There was no significant difference between the curves for dyeings (i) and (ia), (ii) and (iia), (iii) and (iiia), which shows that the rise in pH in normal metachrome dyeing is due to the mordant rather than to the dye, and confirms the dyeing mechanism mentioned earlier.

A further series of dyeings was then carried out to check the above results, and all confirmed the fact that diethyl tartrate can be most effective in preventing the pH of the dyebath from rising when dyeing is carried out in a completely closed system. Whilst it is true that the addition of gluconic lactone will achieve similar results, diethyl tartrate is preferred because it does not give the preliminary quick drop in pH that is produced by the lactone, and consequently there is no risk of throwing the dye out of solution or of obtaining uneven results.

An examination of the physical properties of wool dyed with and without addition of diethyl tartrate to the dyebath was then undertaken, the results being shown in Table I.

Differences exceeding 1.1 oz. are significant for the breaking load and differences of 3.3% are significant for the extension. Whilst the differences between the dyeings produced with diethyl tartrate and those produced without it are generally in favour of the addition, such differences are too small to be significant.

All the foregoing tests were carried out on single hanks which may have come from different windings, and it was felt that hank-to-hank variation

The wool from these dyeings was then tested for breaking strength and elongation (Tables III and IV).

TABLE I

(Samples conditioned and tested at 65% R.H. and 20°C. with 20 in. specimen lengths, constant rate of tensioning 6 in./min., and range 32 oz. Liquor ratio 30 : 1. 20 tests in each experiment)

Ex-periment No.	Dye-ing Con-ditions*	Diethyl Tartrate (%)	Breaking Load Mean Coefficient of Variation (%)	Extension (%) Mean Coefficient of Variation (%)
Undyed wool	...	15.59	7.54	28.83
5% METACHROME BROWN B				
1	Open 0	12.31	10.24	20.28
1 A	Open 2.5	12.91	7.47	21.89
2	Closed 0	13.94	10.39	24.06
2 A	Closed 2.5	14.11	10.06	23.53
5% METACHROME BLUE 8RL				
3	Open 0	14.13	9.41	23.69
3 A	Open 2.5	13.85	11.17	23.11
4	Closed 0	12.28	12.27	22.14
4 A	Closed 2.5	13.89	9.78	21.99

* Open beaker, or closed system under reflux

could easily account for differences as great as those shown. Consequently a further series of trials was arranged in which the dyeings were all done in duplicate and the wool yarn used for all the trials was taken from one large hank. The wool used in these and all subsequent experiments was 4/16 scoured Botany yarn. This series included dyeings of an acid dye both from an acetic acid bath and from a sulphuric acid bath. It was also decided to increase the diethyl tartrate from 2.5 to 7.5% on the weight of the wool in order to see whether this would produce any greater differences in the physical properties of the latter.

The series consisted of the dyeings, all carried out under reflux in a closed system, indicated in Table II. The material used for the experiments consisted of 15-g. hanks of scoured wool yarn all taken from a 1-lb. hank. Before being dyed this wool was wetted out in a 0.4% solution of Teepol (Shell) for 1 min. at 50°C. (liquor ratio 30 : 1), and then washed three times in water. Two of these 15-g. hanks were then set aside as controls. The initial and final pH values of the dyebaths are shown in Table II.

TABLE II

Experi-ment No.	Dyeing*	Diethyl Tartrate (%)	First Dyeings Initial pH	First Dyeings Final pH	Second Dyeings Initial pH	Second Dyeings Final pH
10	(Water extract of) scoured (washed and rinsed) wool	...	7.85	8.02
5	5% Metachrome	0	8.05	8.81	8.02	8.78
5 A	Brown B	7.5	7.09	6.78	7.09	7.00
6	5% Metachrome	0	8.01	8.92	8.00	8.84
6 A	Blue 8RL	7.5	6.95	6.95	7.29	7.01
7	5% Metachrome	0	8.04	8.92	7.97	8.85
7 A	Brown 6G	7.5	6.98	6.71	6.74	6.74
8	3% Wool (+1% CH ₃ COOH)	4.42	5.13	4.42	5.19	...
9	Red 2B (+3% H ₂ SO ₄)	2.18	2.83	2.18	2.02	...
Average		...	8.01	8.85 (+0.84)	7.02	6.88 (-0.16)

* All metachrome dyeings in presence of 5% metachrome mordant + 0.5% ammonia (25%). Wool Red 2B dyed in presence of 30% Glauber's salt (Experiments 8 and 9).

TABLE III

Breaking Loads (Dry)

(Yarns tested at constant rate of loading of 4 lb./min. after conditioning at 65% R.H. and 20°C. Number of tests in each experiment 50)

Experi-ment No.	Mean Breaking Load (lb.)	Coefficient of Variation (%)
10	3.20 3.42 3.31	7.6 5.1 6.0
5	3.38 3.50 3.44	4.1 4.5 4.3
5 A	3.45 3.36 3.40	5.1 4.6 4.8
6	3.33 3.38 3.36	6.1 4.4 5.3
6 A	3.22 3.34 3.28	4.7 5.4 5.0
7	3.18 3.39 3.28	5.0 4.4 4.7
7 A	3.30 3.44 3.37	4.7 6.1 5.4
8	3.26 3.34 3.30	5.2 5.4 5.3
9	3.35 3.26 3.30	4.8 4.5 4.7

D.F.* Variance

Between treatments ...	8	0.0514 (not significant)
Between duplicate samples ...	9	0.0830

* Degrees of freedom.

The differences between treatment means are thus not really any larger than those between duplicates of the same treatment. There is thus no evidence of real differences between means of different treatments.

TABLE IV

Breaking Extensions (Dry)

(Number of tests in each experiment 50)

Experi-ment No.	Mean Extension (%)	Coefficient of Variation (%)
10	31.8 32.2 32.0	8.3 6.2 7.3
5	33.7 33.0 33.3	6.6 7.5 7.0
5 A	34.1 32.6 33.4	8.0 9.7 8.9
6	32.2 32.3 32.2	8.3 7.2 7.7
6 A	31.5 29.9 30.7	8.4 7.8 8.1
7	30.6 32.0 31.3	9.5 8.6 9.0
7 A	31.1 31.1 31.1	8.4 7.3 7.9
8	29.6 27.2 28.4	8.6 9.5 9.0
9	32.5 29.3 30.9	7.8 8.5 8.2

D.F. Variance

Between treatments ...	8	4.625 (significant at 5%)
Between duplicate samples ...	9	1.279

In this case the differences between treatment means are significantly larger than those between duplicates. There is some evidence of real differences between the extension figures of the various treatments. If, however, the analysis is made between the six metachrome dyeings, omitting the acid wool dyeings, then we obtain the following analysis—

D.F. Variance

Between undyed and metachrome dyeings...	1	0.00 (not significant)
Between duplicate samples ...	9	1.279

There is thus no evidence of real difference between the undyed and the metachrome-dyed samples. If we now compare the undyed and acid-dyed samples we obtain—

D.F. Variance			
Between undyed and acid dyeings	1	7.4107	(significant at 5%)
Between duplicate samples	9	1.279	

There is evidence of a difference between the undyed and the acid-dyed samples, but an examination of the figures reveals that quantitatively the difference is small.

A similar set of tests was then carried out comparing wet strengths and wet elongations (Tables V and VI), as it is known that such tests tend to throw into relief any differences in chemical degradation rather more than the dry tests.

TABLE V
Breaking Loads (Wet)

(Breaking load of wet yarn tested at constant rate of loading of 4 lb./min. after wetting out. Number of tests in each experiment 20)

Experiment No.	Mean Breaking Load (lb.)			Coefficient of Variation (%)		
	1	2	Mean	1	2	Mean
10	3.05	2.95	3.00	6.0	4.7	5.4
5	2.78	2.84	2.81	6.1	7.7	6.9
5 A	2.71	2.68	2.69	5.4	1.5	3.4
6	2.61	2.69	2.65	3.4	5.2	4.3
6 A	2.55	2.72	2.64	4.6	4.4	4.5
7	2.57	2.66	2.62	4.4	6.6	5.5
7 A	2.65	2.70	2.67	6.7	4.7	5.7
8	2.74	2.80	2.77	5.4	4.8	5.1
9	2.78	2.80	2.79	4.0	4.8	4.4

Differences exceeding 0.13 between means of breaking load are significant.

TABLE VI
Breaking Extension (Wet)

(Number of tests in each experiment 20)

Experiment No.	Mean Extension (%)			Coefficient of Variation (%)		
	1	2	Mean	1	2	Mean
10	50.9	49.8	50.3	1.5	2.6	2.1
5	49.7	50.5	50.1	3.2	3.3	3.3
5 A	48.8	49.5	49.1	3.1	1.8	2.4
6	48.0	49.8	48.9	1.3	2.3	1.8
6 A	48.1	48.6	48.4	3.1	2.1	2.6
7	48.7	49.7	49.2	3.0	3.7	3.3
7 A	48.0	48.8	48.4	2.9	2.9	2.9
8	50.8	50.6	50.7	2.9	1.8	2.4
9	50.0	49.4	49.7	2.1	2.9	2.5

Differences exceeding 1.5 between means of extension are significant.

A statistical examination of Tables V and VI shows that, whilst there is a significant difference between the wet breaking loads and extensibilities of the undyed wool yarn and the dyed samples, there is no difference between the metachrome-dyed samples and the acid-dyed samples. Also the magnitude of the difference between the undyed wool and the dyed samples is comparatively small and is no greater than would be expected from any

well controlled dyeing process. The results of the statistical examination of these experiments for evidence of difference between the yarns after the various treatments is summarised in Table VII, which is based on significance at 1%.

TABLE VII

	Dry		Wet	
	Break- ing Load	Extens- ibility	Break- ing Load	Extens- ibility
Undyed versus metachrome	No	No	Yes	No
Undyed versus acid	No	No	Yes	No
Metachrome versus acid	No	Yes*	No	No

* In this case the acid-dyed are slightly more affected than the metachrome-dyed samples.

The results of these experiments show that, within the range of pH values which have been experienced in this work, the upper limit of which is pH 9, and with the liquor ratios we have used, there is no evidence that the metachrome process causes any greater damage to the wool, even when carried out in a completely closed system under reflux conditions, than would occur in any other normal dyeing process, and there is some evidence to show that the loss in strength produced by the metachrome process may be less than that produced when dyeing from a sulphuric acid bath.

It was felt that these results should be rechecked by a further series of dyeings using 8% of dye and 8% of metachrome mordant in order to see whether really heavy dyeings made any substantial difference to the wool. This was done using the dyes Metachrome Brown B and Metachrome Blue 8RL. The results obtained were substantially the same as the previous ones and fully confirmed the earlier observations. It is realised that strength and elongation tests probably do not indicate fully the whole effect of the dyebaths on the wool, but it is probable that they do give a very good indication as to whether any significant damage to the wool has occurred.

It has been shown¹² that when wool is damaged by customary chemical treatments its isoelectric point is lowered. Similarly it was felt that, if any difference existed between wool dyed by the normal metachrome process and wool dyed with the addition of diethyl tartrate, this difference might be reflected in the isoelectric points of the wools. Four samples of the same bulk of scoured wool were subjected to the following treatments—

(a) Well washed in running tap water, squeezed, and air-dried.

(b) Dyed according to the standard recipe with 5% Metachrome Brown B in a closed system, well washed in running tap water, squeezed, and air-dried.

(c) As in (b) but with the addition of 2.5% of diethyl tartrate to the dyebath.

(d) As in (b) but the dyeing done in an open vessel.

The isoelectric points of the samples were then determined as follows. Duplicate samples of wool (1.0 g.) were placed in 2-oz. wide-mouthed bottles

with 50 ml. of each of the following solutions—0.0002 N., 0.0001 N., and 0.00005 N. hydrochloric acid, distilled water, and 0.0001 N., 0.0005 N., and 0.001 N. sodium hydroxide. A 10% sodium chloride solution (1.0 ml.) was added to each of one set of bottles, the bottles were sealed with rubber stoppers, and the solutions allowed to stand for two days at room temperature to come to equilibrium. The pH of each solution was then measured. The change in pH produced by the addition of sodium chloride was plotted against the pH of the equilibrium solution in each case, and a straight line drawn through the points. The isoelectric point is clearly given by the intersection of this line and the axis corresponding to zero change in pH, for at this pH neither acid nor base is absorbed preferentially¹³.

TABLE VIII

Solution used HCl (N.)	NaOH (N.)	pH		Δ pH
		Equilibrium Solution only	Equilibrium Solution and Salt	
0.0002	—	3.71	4.81	+ 1.10
0.0001	—	4.67	5.51	+ 0.84
0.00005	—	5.75	5.90	+ 0.15
Distilled water	—	6.52	6.30	- 0.22
—	0.0001	6.50	6.35	- 0.15
—	0.0005	6.88	6.67	- 0.21
—	0.001	7.77	7.00	- 0.77

As a typical example, the values obtained for the washed but undyed wool (Table VIII) indicate that the isoelectric point of the undyed wool is at pH 6.2, those of the dyed samples being given in Table IX.

TABLE IX

Wool	Isoelectric Point (pH)
(a) Undyed	6.2
(b) Dyed in closed vessel	6.8
(c) Dyed in closed vessel and diethyl tartrate added...	6.4
(d) Dyed in open vessel	6.3

The accuracy of the estimation of the isoelectric point of the wools is of the order of ± 0.1 pH unit. Thus, while there may not be any significant difference between the isoelectric points of wools (a), (c), and (d), the isoelectric point of wool (b) is certainly different from them.

It is not known whether any correlation exists between the isoelectric points of wools and their strengths, but this is indicated from these results if it can be assumed that wool is weakened when it is dyed in a closed vessel. In the same way it could be argued that the addition of diethyl tartrate to the closed dyebath would result in the dyed wool having similar properties to wool dyed in an open vessel.

Reference has been made particularly to the use of diethyl tartrate in the experimental work described, but there are other esters which can be used by this technique, examples being phenyl acetate, diethyl fumarate, diethyl malonate, ethyl lactate, and diethyl succinate. The choice of ester to be used is influenced by several factors. It is, however, preferable to use one which is sufficiently

water-soluble to obviate the need for dispersing or emulsifying agents, and diethyl tartrate is eminently suitable in this respect, being completely soluble in water. Ethyl lactate is also suitable, and it may even prove to be slightly better than diethyl tartrate. Both these products are available commercially.

The technique described provides a simple method of controlling the pH in metachrome dyebaths used in closed systems, and of preventing any rise in the alkalinity of the dyebath during the course of dyeing. Since in these experiments no abnormal degradation was observed in the wool samples dyed by the normal metachrome process, it was not possible to show that any advantage was to be gained in this respect by preventing the pH of the dyebath from rising. It must, however, be remembered that bulk-dyeing conditions often differ from laboratory conditions. For example, dyeing times are often longer, liquor ratios vary widely, and different qualities of wool react in different ways, so that control of the pH of the dye liquor under bulk conditions may prove to be more advantageous than it has been possible to show under laboratory conditions. Certain other advantages, however, were observed, for dyeings produced in baths to which diethyl tartrate had been added were generally more level and fuller in appearance than those which had been dyed in a normal metachrome bath.

These results with metachrome dyebaths suggested that perhaps some advantages might be gained by adding diethyl tartrate or ethyl lactate to dyebaths containing the acid milling type of dye and Carbolan dyes. Many of these are difficult to level, and great care must be taken in dyeing them, special techniques often having to be used. Control of temperature and the use of restraining agents have been advocated as methods of overcoming this difficulty. It appears, however, that generally a compromise has to be made between level dyeing and good exhaustion of the dye from the bath. A few dyes were selected, which were known to be difficult to level, and comparative trials were made using the Carbolan Salt A (ICI) method and a method in which an addition of ammonia and diethyl tartrate or ethyl lactate was made to the dyebath. The dyes used were—1% each of Carbolan Fawn RS (ICI), Carbolan Brilliant Green 5GS (ICI), and Coomassie Red PGS (ICI).

The dyes were dissolved with the addition of 0.5% of ammonia (25%), and 2% of diethyl tartrate was added to the dyebath at the commencement of dyeing. The percentages of material used relate to the weight of wool to be dyed, and liquor: material ratios of 100:1 and 60:1 were employed. The results obtained showed that equally good levelling was obtained by this method with rather better exhaustion of the dyebath. This work is still in its early stages, and the specific amounts of ammonia and ester to be added to the dyebath in order to obtain the best results require further investigation. For example, it is known that the acid milling and Carbolan dyes can be classified according to their exhaustion and levelling properties from a neutral dyebath, and

the optimum pH required to obtain the best results varies. Therefore, it may be necessary in some cases to omit the ammonia addition recommended, whilst in other cases it may have to be increased. The amount of ester used may also have to be varied according to the dyes employed, in order to achieve the best results. The results obtained up to the present appear to be very promising, and the method is simple in that no further additions require to be made during the course of dyeing, and a very gradual reduction in the pH of the dyebath is achieved evenly throughout the system by the hydrolysis of the ester.

A further possible application of this method is in the dyeing of some of the direct dyes which are difficult to exhaust by normal methods when applied to cotton and regenerated cellulose rayons, and this aspect is being investigated at present.

A patent application has been filed covering the application and use of these methods and substances in dyeing.

The authors are indebted to Dr. W. Cule Davies for his help and interest in this work, to the Wool Industries Research Association for carrying out the strength and elongation tests on the dyed samples and for the statistical analysis of the results obtained, and to the Directors of Messrs. Brotherton & Co. Ltd. for permission to publish this paper.

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Discussion

Mr. F. MANCHESTER: What is the resulting fibre levelness of dyeings carried out in the presence of diethyl tartrate?

Mr. HANNAY: Up to the present no special investigation of fibre levelness, as distinct from general levelness of dyeing, has been undertaken. It has been found that one of the main differences between the Carbolan Salt A (ICI) method and our proposed method using diethyl tartrate or one of the other recommended esters in the dyeing of acid milling types of dye is that the rate of strike of the dye is considerably lower by our process, although the final depth at the end of dyeing is equal to, and in some cases better than, that obtained with Carbolan Salt A. The general appearance of the

dyeings we have done as regards levelness is in favour of our process.

Mr. I. D. RATTEE: How is it proposed to make shading additions to Carbolan and Coomassie (ICI) dyebaths when the diethyl tartrate method has been used?

Mr. HANNAY: Shading additions would be made in the same way as in most other dyeing processes, depending on the type of dye used for shading. In some cases no readjustment of the dyebath would be necessary, but in other cases, if the final pH of the dyebath were too low to ensure level dyeing of the shading dye to be added, then some readjustment might be required, which could be brought about by adding a small percentage of ammonia to the bath before adding the dye.

Mr. RATTEE: The lecturer has stated that the dyebath goes acid. Does this not mean that the dye added will very rapidly exhaust on to the acid wool?

Mr. HANNAY: It is not strictly true to say that the dyebath goes acid. The effect of the hydrolysis of the ester, which gradually produces acid, is to reduce slowly and evenly the pH of the dyebath during the dyeing time, but the final pH attained depends on the amount of ester used, the initial pH of the dyebath, and the nature of the reaction during dyeing. Thus the conditions may be adjusted to finish with an acid, neutral, or slightly alkaline bath according to the conditions required for the type of dye being applied.

Dr. J. F. GAUNT: Our experiments on the use of diethyl tartrate with fast acid dyes show that this agent alone will not produce satisfactory exhaustion with many dyes. It is necessary to add either acid or Glauber's salt to exhaust the bath. What effect have these agents on the rate of hydrolysis of diethyl tartrate?

Mr. HANNAY: The work described in this paper has been restricted to an investigation of the dyeing of metachrome, acid milling, and Carbolan dyes, all of which exhaust well under neutral or slightly alkaline conditions, but which often give difficulty in respect of levelling. It has been realised that quite a number of dyes require more acid conditions to attain good exhaustion, and further work is being carried out to investigate such a system. Hydrolysis of the esters proceeds quite evenly and well down to a pH of 5, and the presence of salts such as Glauber's salt, ammonium acetate, or ammonium sulphate does not interfere with the reaction. It has also been found that some dyes exhaust well at slightly higher pH values by our method in the presence of such salts, and work is continuing along these lines.

Dr. GAUNT: We have found that the rate of exhaustion of Coomassie Fast Scarlet BS during the first 30 min. of dyeing, during which the temperature was raised from 40°C. to the boil, is much lower with 2.5% diethyl tartrate than with 1% Carbolan Salt A and 20% Glauber's salt, but that the final exhaustions after 1 hr. boiling are practically identical. This agrees with the results shown by Mr. Hannay.

Some Developments in the Setting and Finishing of Nylon Textiles

G. K. MECKLENBURGH, S. SHAW, and H. W. PETERS

Meeting of the Midlands Section held at the King's Head Hotel, Loughborough, on 23rd April 1952, Mr. A. P. Kershaw in the chair

The conditions required for setting nylon textiles both by steam under pressure and by high-temperature dry heat are described. The methods available to the finisher are reviewed, and particular attention is given to possible advantages and disadvantages. The use of infrared radiant heating as an alternative to hot gases for the high-temperature dry heat method is also discussed. A comparison is drawn between the properties of nylon fabrics set by these alternative methods, and this is followed by an outline of the results of some investigations into the use of swelling agents during dry heat setting. The final section deals with a new method of permanently stiffening nylon fabrics by impregnation with inorganic salts, zinc chloride in particular, followed by heating under controlled conditions.

1. INTRODUCTION

It is only a few years since the application of nylon in the textile industry of this country was limited to the hosiery field. Experience fully justified the confidence which had been placed in this new fibre, which was different in several respects from all other known fibres. A high yarn strength and good elasticity, combined with excellent wearing properties, placed nylon as the undisputed hosiery yarn. Its success in this field suggested greater possibilities, such as a wider and fuller use in lightweight wearing apparel. Plain-woven nylon parachute fabrics had, however, demonstrated the lack of several desirable characteristics, such as a good handle and drape, and indicated that any progress in this field of lightweight wearing apparel would depend upon fabric construction, especially if a fabric possessing a soft and appealing handle was to be developed. As a result, alternative woven and knitted constructions were examined, and it was early recognised that each offered wide but different possibilities. Nylon warp-knitted fabric, or tricot, made an immediate appeal as a lingerie material in lightweight locknit and fancy-mesh constructions having a high strength and a soft handle. Woven fabrics, on the other hand, offered interesting possibilities in voile, seersucker, and similar constructions, providing high strength with lightness and serviceability, but possessing a certain attractive crispness in handle and appearance.

Throughout this initial development period, when, because of increased availability, nylon was expanding into new end uses, a new and important factor was also receiving attention and its fundamental importance was rapidly becoming recognised. It was shown that the character of nylon fabrics, both woven and knitted, could be developed and improved by the proper application of the setting process—the process which had been shown to be vital in the finishing of nylon stockings, and without which no satisfactory stocking could be produced. Applied to plain-woven "paranylon", a definite improvement in handle could be effected by the setting process. This conferred on the fabric the important property of dimensional stability and resistance to permanent creasing during scouring and dyeing, or other hot wet processes such as hand washing of made-up garments. Warp-knitted nylon fabrics derived even greater benefit from being set, owing to their

inherent instability and pronounced edge-curling tendencies. For these knitted fabrics, in particular, the setting process has made possible new standards of quality of finish and consumer performance. But to whatever use nylon yarn is put, it is essential to consider the extent to which a setting process is necessary or desirable, and for this reason a close study of the behaviour of nylon yarn and fabrics towards setting conditions has been made.

2. SETTING CONDITIONS

A previous publication¹ has dealt in some detail with the behaviour of nylon yarn and fabric towards both pressure steam and high-temperature dry heat, and suitable setting conditions for use with woven and knitted fabrics have been established.

The use of pressure steam as a setting medium is so well established, that little further comment is required. Provided that complete control can be exercised over the fabric, the excellent stabilising influence of steam at 120–140°C. (corresponding to an excess pressure of 15–35 lb./sq. in.) encourages universal acceptance of this method. The exclusion from the steam atmosphere of all products which are capable of reacting with the nylon makes the conditions safe to use, so that the finisher can decide the time of steaming required to ensure uniformity of treatment. In the absence of restraint, nylon yarn will shrink on exposure to these steaming conditions to an amount depending upon its previous thermal history. The difficulty of maintaining control over the fabric dimensions has restricted the wide application of steam setting for all types of woven and knitted nylon fabrics.

The success which can be achieved by using high-temperature dry heat setting conditions is well demonstrated by the existence of several machines in this country, and many more elsewhere, which incorporate a dry hot zone and which are producing well set nylon fabrics. With this method, alternative sources of heat, such as hot gas and infrared radiation, may be used; but, compared with steam setting, much less latitude is placed in the finisher's hands owing to the need for very careful and accurate control of temperature, time of processing, and condition of the fabric. The advantages of the method will be discussed in more detail later, but in view of the need to emphasise the importance of very close control of operating conditions a

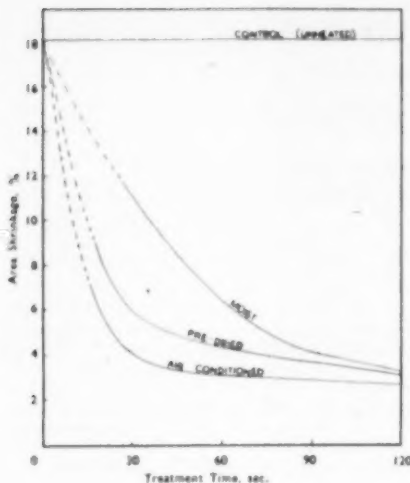


FIG. 1—370°F.

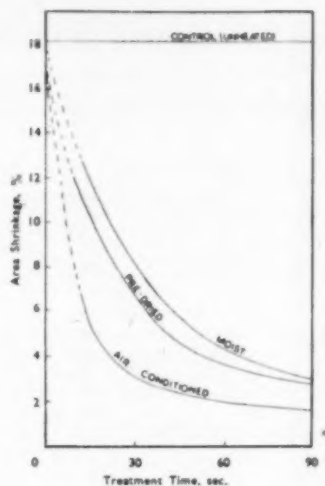


FIG. 2—385°F.

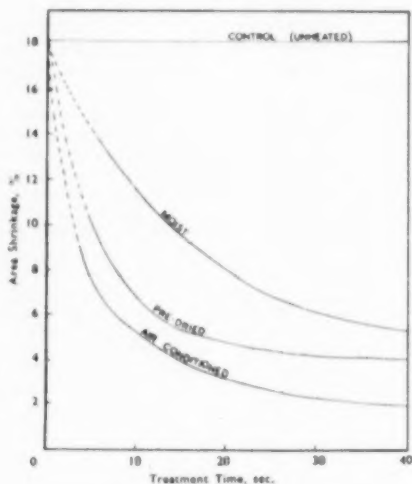


FIG. 3—405°F.

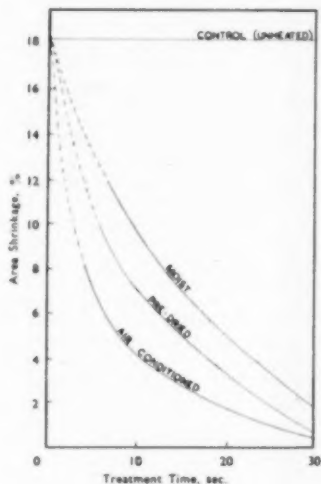


FIG. 4—420°F.

Fabric held to dimensions during heat treatment

FIG. 1-4—Effect of Air Temperature in Dry-heat Setting

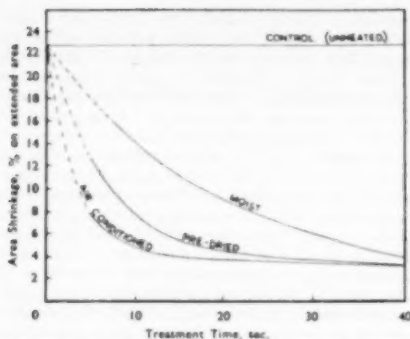
summary of the factors involved in this method is opportune.

Up to the present time hot gas conditions have been used for this study, and Fig. 1-6 summarise the relationship which has been found in the laboratory to exist between temperature of treatment, time of treatment, and moisture condition of the fabric. The investigations were carried out on

loom-state locknit fabric constructed from 30/10/18 nylon yarn¹.

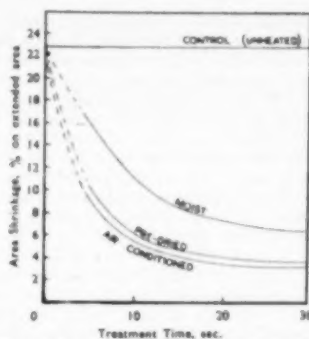
A number of interesting features are evident from these graphs.

The presence of a small amount of moisture (regain moisture) assists the setting process. It is considered that this moisture exerts a swelling action which, together with the influence of heat,



Fabric extended 3% lengthwise and widthwise during heat treatment

FIG. 5—Air Temperature 405°F.



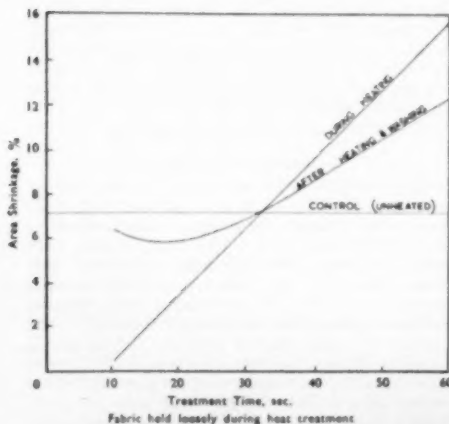
Fabric extended 3% lengthwise and widthwise during heat treatment

FIG. 6—Air Temperature 420°F.

permits structural rearrangement and increased stability of the nylon molecular chains. A large amount of moisture retards the rate of heating of the fibres.

The graphs also show that a greater degree of set is imparted to the fabric as the temperature is raised, provided that the heating time is maintained constant. This applies to fabric which is held to dimensions or held slightly extended in length and width.

The time of treatment may be varied by adjusting the temperature accordingly within the approximate limits of 370–450°F. (188–232°C.), but it should be clearly recognised that the time of heating a nylon fabric in a hot atmosphere containing oxygen is strictly limited because of the thermal oxidative degradation which rapidly takes place. The use of a high temperature for a short exposure time has been found to be less liable to cause yellowing than setting at a lower temperature for a longer period of time; this increases the practical difficulties during processing.



Fabric held loosely during heat treatment

FIG. 7—Air Temperature 430°F.

Fig. 7, which was obtained by tests on scoured locknit fabric constructed from 30/10/18 nylon yarn, illustrates the fact that tension is an integral part of the setting process. Little or no dimensional stability is imparted to a fabric held loosely when exposed to the hot atmosphere, and, by prolonging the heating time at a constant air temperature, the fabric shrinkage becomes greater than the maximum shrinkage of an unheated sample when submitted to the boiling water test*. In fact, subsequent washing causes an extension in area owing to the swelling action of imbibed moisture.

If infrared heating is adopted, a similar approach is necessary in order to establish effective setting conditions and determine the degree of latitude which can be tolerated.

The dry heat method, which makes use of a hot roll, the nylon fabric being set by controlled contact with the heated surface, is also a successful means of stabilisation and is being applied in the setting of both woven nylon fabrics, broadloom and ribbons, and also knitted fabrics. Here, again, similar experimental work is necessary to determine optimum setting conditions.

3. SETTING METHODS

Steam Setting

Steam setting has been practised for many years, and finishers have been able to produce fabrics of excellent stability and a full soft handle. The principle of using pressure steam in an enclosed vessel as a setting medium is well known, and the fact that it is a batch process is not necessarily a disadvantage, since this will depend both on the ancillary capacity in the dyehouse and also on the volume of business handled. The uniform control of fabric width is not a simple matter, and the batch of fabric requires careful preparation before placing in the autoclave.

It is not usual to set nylon fabrics in the grey state by this method, since any contamination will be set into the nylon to such an extent that it becomes extremely difficult to remove by normal scouring techniques. Moreover, the high potential

* 30 min. immersion in boiling water¹.

fabric shrinkage prevents adequate control of the fabric either to a specified width, or to a uniform width from end to end of the piece on the batch roll. An initial scour, usually at a low temperature, is, therefore, normal practice, not only to remove oil, size, and any dirty marks, but also to reduce the shrinkage factor, thereby placing more control in the hands of the finisher. The absence of direct positive dimensional control of the fabric is regarded as a big disadvantage with this method. Moiré effects are also sometimes produced where the fabric shrinks tightly on itself or on the interleaving blanket cloth. Moreover, whilst it is essentially true that each knitted fabric construction has an optimum finished width, it is more important with this method, than with the dry heat method, to prepare the batch at or near to this width. If too wide a batch is made, high shrinkage will result during steaming. This is unlikely to be uniform and would then lead to unevenness in subsequent dyeing. Despite these drawbacks this method can be successfully operated, but the finisher must be allowed some tolerance in meeting a required width and, even then, this width should lie close to the optimum finished width to ensure the production of a stable fabric.

Dry-heat Setting

Both gas and infrared heating are used, always in conjunction with a stenter frame for accurate control of fabric dimensions during the heat treatment. Owing to the positive control it is possible to set the fabric in a stretched condition, thereby obtaining a high yield. In some cases the finisher endeavours to obtain the very highest yield from the fabric, consistent with reasonably satisfactory stability, in order to meet his customer's request. It is, however, a much more difficult task to stabilise nylon fabric to meet the boiling water test when high yields are demanded. It would be far better to seek first and foremost quality of finish and complete stability. For high yields, setting conditions have to be slightly more severe in order not only to eliminate yarn shrinkage, but also to stabilise a stretched fabric structure and avoid subsequent structural relaxation. Severe setting conditions reduce the margin of permissible control limits, and all three conditions of temperature, time, and fabric dimensions have to be very carefully balanced.

The importance of temperature recording and control cannot be too strongly emphasised whatever method of heating is used.

Hot-gas Setting

The exposure of a controlled web of nylon fabric for a specified length of time to an inert atmosphere maintained at constant temperature, in order to raise the temperature of the nylon yarn quickly to that temperature and then subsequently to hold it at that temperature until suddenly cooled, represents almost ideal setting conditions. In practice, the method taxes the ingenuity of the machine manufacturer in the design of the machine and the skill of the finisher in operating the machine and handling the cloth. The ability to hold the fabric at a precise temperature for as long as

required is a very good feature indeed and a great advantage. Uniformity of temperature across the fabric web depends on the design of the heating system but is obviously a prime requisite for successful setting. Non-uniform heating causes irregular stability and variable dyeing affinity. These troubles also arise if the fabric is presented to the setting zone in a non-uniform condition of moisture content. In certain cases, where cleanliness of the fabric permits, setting in the grey state may be carried out on the hot gas stenter; otherwise, fabrics should be scoured first and then dried, preferably in a separate housing on the same stenter frame. Alternatively, the fabric may be presented to the setting machine uniformly damp, and the setting time adjusted to allow for evaporation of the moisture. Adequate provision should be made to cool the fabric properly before batching, otherwise the fabric may remain hot for quite long periods, thus causing degradation, discoloration, and differences in dyeing affinity and handle from batch to batch.

The method of hot-gas setting suffers, however, from certain handicaps. A stoppage of the machine will result in cloth damage owing to overlong heating. Yellowing of the fabric occurs to a small extent, owing to the difficulty of rigidly excluding oxygen, and post-setting of dyed goods has not been successfully practised. Besides yellowing of the dyed shade the hot products of coal-gas combustion, which are generally employed, may also have a destructive action on the dyes used. The system is also susceptible to fluctuations in coal gas supply. The housing, furthermore, is a very expensive item, but there is no doubt that under proper operating conditions nylon fabrics can be given a very high-quality finish on a hot-gas setting stenter.

The problem of yellowing can be tackled in two ways. Firstly by the use of antioxidants, which are themselves preferentially oxidised and which can be applied to the fabric during the initial wet processing before setting; and secondly by removing the discoloration by bleaching with sodium chlorite in acid solution. Since only a very small proportion of oxygen is necessary to produce discoloration at very high temperatures, fully inert hot gases (e.g. nitrogen and superheated steam) for setting are clearly desirable, but these conditions may not be easily or cheaply attained.

Radiant-heat Setting

This method is based on the use of infrared radiant heat units and is a method gaining in popularity, especially in America. The fabric to be set is fed continuously to the stenter frame, over which is mounted the radiant heat assembly. This takes the form of units comprising elements which, when heated electrically, emit infrared radiation of wavelengths strongly absorbed by the nylon. One such unit is made by Sill Industries of New York, and the Fiberglas Super heater is composed of nichrome wire elements embedded in a matrix of glass fibres. These units, of which two or more are normally required, are operated at a recommended surface temperature of 700°F.

(370°C.) for nylon setting, and it is therefore necessary to adjust the distance of the unit from the cloth surface. The temperature may, however, be varied to suit fabrics of different weights and types.

A more recent unit has been developed by the McCreary Machine Works Inc., Cohoes, N.Y., in conjunction with the General Electric Co. of America. G.E. Calrod tubular heaters provide the source of heat, and these are housed in high-temperature alloy sheaths which are themselves mounted in reflecting frames. Like the Sill unit, these frames are designed to be raised automatically if the flow of fabric through the machine stops for any reason, and thus to prevent scorching. The Calrod tubes are set at a slight angle to the direction of fabric movement, thereby ensuring completely uniform heating of the fabric from edge to edge. As with the gas setting method, particular attention has been paid to the accurate control of temperature, and automatic temperature control is provided by an electronic control unit operated by thermocouples located in the reflector frames of the heating units.

In this country the General Electric Co. Ltd. and the British Thomson-Houston Co. Ltd. manufacture infrared heating elements of a similar type to the G.E. Calrod tube, which can be utilised in a similar way in conjunction with a stenter frame.

Radiant-heat setting finds favour for a number of reasons. The process can be controlled by the finisher and a stoppage of the machine does not result in damaged cloth; the housing required is inexpensive, and is necessary, not for insulation purposes, but solely to exclude draughts; the units are, moreover, readily adaptable to existing stenter frames. The McCreary unit has easily replaceable parts, and maintenance is simple. The Sill unit is not accessible in this respect, but it is of robust construction and with care should have a life of several years. Radiant heat is "clean" heat and permits post-setting both of dyed and of undyed fabrics, causing virtually no discoloration of the set fabric. Availability of oxygen for yellowing is limited on account of the relatively still atmospheric conditions prevailing around the heating units compared with the high-velocity circulating system employed in the hot gas method, and also an important contributory factor in reducing any yellowing tendency is the much shorter setting time employed for radiant heat setting. Here, again, provision should be made to cool the fabric immediately after setting. A scoured and bleached locknit fabric can be post-set by radiant heat without further treatment.

The use of such a technique as this requires even more critical control of the operating conditions than is demanded by the hot gas method. The chief reason for this is the very rapid rise in temperature of the nylon under efficient operating conditions and the complete absence of any period of dwell at the setting temperature. Previously dried fabric should be presented to the infrared radiation, because water vapour strongly absorbs over the same wavelength ranges as the nylon. The predrying should be controlled to leave approximately regain moisture content in the

cloth, since practical experience has indicated that this is very beneficial, and this confirms the results of our own study with a hot gas medium.

4. A COMPARISON OF THE PROPERTIES OF FABRICS SET WITH DRY HEAT AND PRESSURE STEAM

In view of the availability of alternative methods of setting, it was natural to enquire further into the subject in order to compare the properties of nylon fabrics set with dry heat and pressure steam. The final character and handle of the fabric had been observed to differ appreciably depending on the method of processing. A cursory examination of these aspects led to the immediate conclusion that the method of setting exerted a very great influence, and needed to be recognised as the largest single controlling factor in developing the final fabric properties. A detailed investigation was therefore carried out to compare these properties with special reference to handle and susceptibility to creasing on subsequent hot wet processing, properties which were considered to be highly important from the finisher's point of view.

A certain number of factors should be pointed out in connection with the experimental conditions used. A hot-air electric oven was used for dry-heat setting, and air movement was slow; the tendency to yellow, therefore, was less than it would be in an efficient machine circulating hot gases at high velocity. The susceptibility to creasing was assessed by immersing the sample in a crumpled condition in boiling water and boiling for 30 min. This represents a really severe test.

An interesting observation made during the trials was that, when the fabric set with dry heat was removed from the setting oven and therefore cooled, an immediate extension in area occurred, so that the fabric became loose and sagged slightly even though it was tight on the frame during the setting process. In contrast to this, fabric set with pressure steam was found to be slack on the frame at the end of the setting process, but on drying contracted considerably in area and became very taut. It should be noted that in both cases the fabric was held under slight tension at the beginning of the setting treatment; it is not known at what stage of the pressure-steam setting process extension in area occurred.

The work was extended in order to examine the effect of cold water quenching immediately after dry heat treatment and also to measure the effect of all the setting treatments on fabric bursting strength and relative viscosity of the nylon.

Table I summarises the results of a series of tests carried out both on plain woven 45/15/308 nylon yarn, 105 ends \times 105 picks, and on locknit (30/10/7½Z nylon yarn) fabrics which were subjected to pressure-steaming conditions, and Table II the results with dry-heat setting conditions.

As a result of all this work it was shown that fabrics set with hot air are, in general, more susceptible to creasing when subjected to boiling water than are fabrics set with pressure steam. The former fabrics do, however, tend to recover from creasing to a certain extent during conditioning (i.e. ageing). This ageing factor is not

uncommon to finished textile fabrics made from other fibres, e.g. crease-resisted spun viscose rayon fabrics.

On the question of handle, it was noted that, in general, a softer and fuller handle is obtained by

TABLE I
Effect of Pressure Steam on Fabric Properties

Steam Pressure (lb./sq. in.)	Steam Temp. (°C.)	Setting Time (min.)	After treating for 30 min. in Boiling Water	Area Shrinkage (%)	Yellowing	Creasing
PLAIN WEAVE FABRIC						
Unset control	—	—	—	7.5	—	Marked
10	115	10	2.2	Nil	Moderate	
		60	2.2	Nil	Moderate	
15	121	10	2.8	Nil	Moderate	
		60	2.8	Nil	Slight	
20	126	10	1.7	Nil	Slight	
		60	2.2	Nil	Very slight	
25	131	10	1.7	Nil	Slight	
		60	1.7	Nil	Very slight	
LOCKNIT FABRIC						
Unset control	—	—	—	13.1	—	Marked
10	115	10	4.4	Nil	Moderate	
		60	4.7	Nil	Slight	
15	121	10	4.2	Nil	Moderate	
		60	4.0	Nil	Slight	
20	126	10	4.7	Nil	Very slight	
		60	3.5	Nil	Very slight	
25	131	10	3.7	Nil	Very slight	
		60	2.8	Nil	Very slight	

TABLE II
Effect of Dry Heat on Fabric Properties

Air Temp. (°C.)	Setting Time (sec.)	After treating for 30 min. in Boiling Water	Area Change (%)	Yellowing	Creasing
PLAIN WEAVE FABRIC					
Unset control	—	—	—	6.8	Marked
174	10	—	5.9	Nil	Marked
	25	—	4.4	Nil	Marked
188	10	—	4.3	Nil	Marked
	25	—	3.8	Nil	Marked
196	10	—	3.1	Nil	Marked
	25	—	0.5	Nil	Moderate
204	10	—	3.5	Nil	Marked
	25	—	0.5	Nil	Moderate
216	10	—	0.7	Nil	Marked
	25	—	0.8	Very slight	Moderate
224	10	—	1.1	Nil	Moderate
	25	—	1.8	Slight	Slight
LOCKNIT FABRIC					
Unset control	—	—	—	13.1	Marked
174	10	—	11.3	Nil	Marked
	25	—	7.5	Nil	Marked
188	10	—	12.9	Nil	Marked
	25	—	8.8	Nil	Marked
196	10	—	12.0	Nil	Marked
	25	—	6.4	Nil	Moderate
204	10	—	10.3	Nil	Marked
	25	—	5.8	Very slight	Moderate
216	10	—	8.1	Nil	Marked
	25	—	4.3	Slight	Slight
224	10	—	6.6	Nil	Moderate
	25	—	2.5	Slight	Slight

pressure-steam setting than by dry-heat setting. The assessment of handle is a matter of personal like and dislike, and is related to the type of fabric and its end use. It is considered that there will be an outlet for both steam-set and dry-heat-set fabrics, though it may well be that dry heat setting processes can be so modified as to impart a variety of qualities of handle to a fabric, especially by an experienced finisher. A larger-scale trial was conducted, and three pieces of fabric—a locknit, an open-mesh tricot, and a voile—were all processed together, except that half of each piece was dry-heat set and the other half steam set. It was generally agreed that the steam-set fabrics possessed the fuller or loftier handle. However, finished length and width measurements of these pairs of fabrics must also be considered in any assessment. In each case the dry-heat-set fabric was greater in area than the steam-set fabric. One can therefore justifiably state that in this particular trial the dry-heat-set fabrics were thinner than the steam-set ones, and this would, in part, account for the difference in handle. In all cases these fabrics had a good appearance, the dry-heat-set samples being more lustrous and sheer than the steam-set ones.

Other points brought out by the investigation are that more stringent conditions, of either steam or dry heat, are required to stabilise a knitted than are required to stabilise a woven fabric to boiling water. There is a slight tendency for water quenching to cause a less pleasant handle, particularly noticeable in those water-quenched samples which exhibit high residual shrinkage, i.e. which are inadequately set. Relative viscosity tests showed that chemical degradation can occur if the dry-heat setting is unduly prolonged, but a proper degree of set can be imparted without causing any chemical damage.

5. THE USE OF SWELLING AGENTS DURING DRY-HEAT SETTING*

Following on from the work already discussed, the use of swelling agents during dry-heat setting has been investigated—a modification which, in the future, may open up a new field for development.

The major differences in the two methods of setting are the swelling action of the steam under pressure and the different temperature levels which are used. The soft full handle and improved resistance to creasing of pressure-steam-set fabrics are thought to be due partly to the swelling action of the steam, although shrinkage effects undoubtedly play a part. It is suggested that the nylon is swollen during the steam setting process, permitting a considerable degree of molecular movement to occur. Combined with the thermal agitation caused by the rise in temperature of the yarn, the overall effect on the fine molecular structure differs from the final result produced when the temperature effect alone is operating, even when the thermal energy applied is much greater. It is feasible that a greater "relaxation" occurs, characterised by more folding, coiling, and reorientation of the chain segments. This will

* Patent protection applied for

certainly happen if the restraint on yarn shrinkage is reduced. If shrinkage is prevented, then the longitudinal orientation of the chains may be altered, and transverse orientation become more pronounced. Whatever the true reason, subsequent hot wet treatments do not superimpose another set on that already imparted by the pressure steam, but it would appear that the setting activity of boiling aqueous liquors is additive, to a certain limited extent, to a dry-heat set. This points to the complementary nature of heat and swelling agents for setting nylon fabrics.

Further investigations, therefore, were planned to study the use of high-boiling swelling agents during dry-heat setting, and the work was extended to cover the use of an electrolyte with the swelling agent.

Some of the swelling agents examined were ethylene glycol, glycerol, benzyl alcohol, phenol, and *m*-cresol; and sodium chloride, sodium thiosulphate, calcium chloride, zinc chloride, magnesium sulphate, and aluminium sulphate were among the electrolytes selected. Samples of scoured nylon locknit fabric constructed from 30/10/74Z yarn were padded with dilute aqueous solutions of the swelling agents, either alone or in conjunction with the electrolytes, and then were dry-heat-set in hot air at 420°F. (216°C.) for 30 sec. whilst held to controlled dimensions on a pin frame. The degree of set was determined by subjecting the samples to a 30-min. treatment in boiling water, the degree of creasing was noted, and the handle of the set and washed samples was assessed qualitatively.

TABLE III
Dimensional Stability of Dry-heat-set Locknit Fabric pretreated with Aqueous Solutions
(Area change, %)

Pretreatment Soln. (% concn.)	5 min. after setting	After setting and conditioning for 4 hr. (1)	After Dimensional Stability Test (2)	Difference between (1) and (2)
Untreated control	... + 1.9	+ 3.6	- 2.2	5.8
1.1% <i>m</i> -Cresol + 1.1	+ 0.6	+ 0.2	0.4
4% Benzyl alcohol + 5% Aluminium sulphate + 1.8	+ 2.3	+ 1.7	0.6
4% Benzyl alcohol + 5% Sodium chloride + 1.1	+ 0.8	- 1.0	1.8
4% Benzyl alcohol + 10% Sodium thiosulphate + 1.8	+ 2.1	+ 1.7	0.4
4% Benzyl alcohol + 10% Calcium chloride + 1.2	+ 1.8	- 0.6	2.4
+ ...	Extension compared with pin dimensions			
- ...	Shrinkage compared with pin dimensions			

Table III illustrates the results of the dimensional stability tests, and shows that the overall dimensional changes of the pretreated samples were less than those of the untreated samples. Moreover, the pretreated fabrics showed an increased resistance to creasing during treatment in boiling water and subsequent dyeing. Some of the fabrics also showed improved crease resistance when tested by hand crushing; this was thought

to be most noticeable in the case of fabric pretreated with a solution of 5% sodium thiosulphate plus 4% benzyl alcohol.

Improved drape and a softer fuller handle, akin to those of pressure-steam-set fabric, was exhibited by fabric pretreated with either a 4% aqueous benzyl alcohol solution or an aqueous solution containing 4% benzyl alcohol and 5% sodium thiosulphate.

A semi-bulk dry-heat setting trial on similar locknit fabric pretreated with an aqueous solution containing 4% benzyl alcohol alone and in conjunction with 5% sodium thiosulphate has been carried out. It was considered that an improvement in handle had been achieved.

A further trial was carried out on a woven fabric, and, as will be seen from the stability results (Table IV), the pressure-steam-set fabric was better set than the others. It was considered that if the samples treated with benzyl alcohol had been equally well set their performance would have been better.

TABLE IV
Results of Laboratory Washing Tests on Modified Dry-heat-set Woven Fabric*

Fabric Treatment	Stability to Boiling Water (%) (shrinkage)	Appearance (creasing, etc.)	
		After 5 Washes at 40°C.	After a Further Wash at 85°C. for 30 min.
(i) Pressure-steam-set (control fabric)	0.6	Very good	Good
(ii) Dry fabric, no pretreatment, dry-heat-set	1.8	Good	Fairly good
(iii) Damp fabric, no pretreatment, dry-heat-set	3.6	Poor (sharp creases)	Very poor
(iv) Pretreated with 4% benzyl alcohol + 5% sodium thiosulphate, dried, then dry-heat-set	2.2	Good	Quite good (superior to (ii), inferior to (i))
(v) Pretreated with 4% benzyl alcohol + 5% sodium thiosulphate, then dry-heat-set in damp state	3.1	Good	Very poor

* FABRIC CONSTRUCTION—

Warp	...	60/20/74S nylon yarn
Weft	...	2/60/30/158 nylon yarn
Ends per inch	...	160
Picks per inch	...	80
Plain weave	...	

The handle of the fabrics was judged by seven observers; and the consensus of opinion was that the pressure-steam-set fabric was slightly superior to the fabric treated with alcohol and thiosulphate and set dry, and the two samples which were not pretreated were considered to be harsh.

6. THE STIFFENING OF NYLON†

In the course of this work on swelling agents it was found that some combinations of swelling agent and electrolyte imparted a stiff finish to nylon fabric on subsequent dry-heat setting. Examples of such combinations were—

- (1) An aqueous solution containing zinc chloride, calcium chloride, cadmium

† Patent protection applied for

chloride, or cadmium nitrate, each in conjunction with benzyl alcohol.

- (2) An aqueous solution of formic acid plus benzyl alcohol.
- (3) An aqueous solution of *m*-cresol, benzyl alcohol, and sodium thiosulphate.

Further work led to the discovery that aqueous solutions of some inorganic salts used alone, e.g. aluminium chloride, stannic chloride, and zinc chloride, produced a stiff finish on nylon when heated. Zinc chloride was selected for a full investigation both in the laboratory and on a works scale, in order to ascertain the practical value of the process. The results have been most promising and have opened up an entirely new field for development.

All the aqueous zinc chloride solutions used in the stiffening trials were acidified with acetic acid in order to take up the precipitate of zinc oxychloride which is formed by hydrolysis; this gave a pH of approx. 5.0-5.5.

The zinc chloride solution may be applied at room temperature by an immersion technique followed by the removal of excess solution. After treatment, the material may be dried at low temperature, and subsequently heated to a higher temperature to develop the desired stiffness. Alternatively, it may be dried and stiffened in one operation. The time of heating is important, since if unduly prolonged, especially when stiffening and setting are carried out as one operation, yellowing occurs and, eventually, yarn degradation.

The stiffening is accompanied by delustring, which is particularly noticeable on heavier-type fabrics when high concentrations of zinc chloride are used. The dull matt effect achieved is in many instances a desirable feature.

In view of the chemical nature of the finish and the modification of the nylon fabric by controlled chemical attack, a whole series of trials was planned in order to establish the possibilities and limitations of the process.

TABLE V
Effect of Zinc Chloride Stiffening on the Strength of 45-denier Semi-dull Yarn

(Set at 420° F. for 20 sec. Exposed to 220 hr. sunlight)

Zinc Chloride (% concn.)	Breaking Load (g.)	
	Unexposed	Exposed
Unset control	236	81
Set control	245	67
1.25	202	91
2.5	181	111
5.0	157	80

The first aspect obviously concerned the effect upon the strength of the yarn itself. Table V shows the average results of some physical tests carried out on normal semi-dull 45-denier yarn which had been treated with zinc chloride solutions of varying concentration and then dry-heat-set at 420° F. (216°C.) for 20 sec. whilst prevented from shrinking. The breaking loads are compared before and after exposure to sunlight for 220 hr. It is shown that an increase in concentration of zinc chloride is accompanied by a reduction in strength of the yarn. However, the relative drop in breaking load of untreated and treated yarns on exposure indicates

that the zinc chloride treatment does not increase the light degradation of the yarn.

As it was found that stiffening occurred when pretreated fabric was dried at most temperatures, some unset locknit fabric constructed from 30/10 7½Z nylon yarn was lightly scoured, then impregnated with a 10% solution of zinc chloride, and dried at various temperatures ranging from 80° to 160° C. (176° to 320° F.) in order to find the effect of the drying temperature on the stiffness. The results are given in Table VI, which also includes the stability conferred on the fabric by stiffening at these temperatures, which are lower than those used for setting.

It will be seen that unset fabric can be stabilised by a zinc chloride stiffening treatment provided the

TABLE VI
Effect of Temperature of Drying on Degree of Stiffness and Dimensional Stability of Locknit Fabric

(10% zinc chloride soln. Progressive increase of stiffness with temp.)

Drying Time (min.)	Drying Temp. (°C.)	Area Dimensional Change after Boiling Water Test and Conditioning (%)
15	80	-6.5
15	98	-7.0
10	116	-7.1
5	140	-3.0
2.5	160	-0.3

drying temperature is sufficiently high; stabilisation, however, is related to the degree of stiffness.

Breaking Load-Extension

For any given concentration of zinc chloride solution, the degree of stiffening is not fully developed until setting temperatures are used, and for maximum stiffness and permanence stiffening at setting temperatures is to be recommended. Naturally, on a lightweight fabric such as tricort or nylon voile, the degree of stiffness attainable by this process is limited on account of the need to avoid undue yarn degradation. This danger is illustrated by the results given in Table VII. They apply to a nylon voile which had been impregnated with a 2% solution of zinc chloride (approx. 40% retention of liquor) and then dry-heat-set at 420° F. (216°C.) for 30 sec. The figures are the mean of six breaking load-extension tests.

TABLE VII
Effect of Zinc Chloride Stiffening on Breaking Load-Extension of Nylon Voile*

	Warp		Weft	
	Un-treated	Treated	Un-treated	Treated
Breaking load, lb.	77.3	54.7	77.1	61.6
Extension, %	38.1	28.1	41.4	32.2

* FABRIC CONSTRUCTION—

Warp and Weft	Constructed from 45/15/30Z nylon yarn
Ends per inch	100
Picks per inch	100

Although appreciable decrease in breaking load and percentage extension at break occurred, the fabric remained quite strong because of the high initial strength of the nylon yarn. The use of a 1% solution of zinc chloride would probably have been

more suitable for this fabric. It is worthy of note that, in view of the tensile strength results obtained on yarn after exposure (Table V), the fabric would be expected to maintain its strength during wear at least as well as untreated fabric, maybe better.

Experience has shown that this degradation factor can be controlled.

Abrasion Resistance

Samples of 30-denier nylon warp-knitted shirting fabric, dyed blue, having great susceptibility to fluffing during wear, were slightly stiffened with 1% zinc chloride solution at 130–140°C. (266–284°F.). Comparative rubbing trials with untreated fabric were carried out on a Martindale wear-testing machine. The results (Table VIII) show an exceptional increase in resistance to abrasion of the treated fabric over the untreated fabric.

TABLE VIII
Effect of Zinc Chloride Treatment on the
Abrasion Resistance of Nylon Warp-knitted
Shirting Fabric

Untreated	Treated
Badly worn at 52 rubs	Very little signs of wear at 52 rubs
Hole worn at 78 rubs	Slight wear at 150 rubs
	Very little change up to 260 rubs
	Very worn in appearance at 520 rubs
	Hole worn at 1389 rubs

As is well known, the abrasion resistance of nylon is generally very good. However, with some yarn and fabric constructions filamentation, or fluffing, readily occurs. This is improved by the zinc chloride process, which fuses the filaments together, and subsequent rubbing is analogous to

abrading a nylon rod rather than a textile yarn composed of many filaments.

Another trial was carried out to illustrate this aspect of abrasion resistance. A few comparative tests were made on nylon webbing pretreated in various ways. Table IX indicates the treatments given and the number of rubs required to produce comparable signs of wear, and from it the advantage in using a zinc chloride treatment is readily seen.

TABLE IX
Abrasion Resistance of Nylon Webbing treated with
Zinc Chloride and Resin Finishes respectively

Treatment of Webbing (% by wt.)	No. of Rubs
Zinc chloride (5%)	56,735
Merlon BR (7.5%)	46,100
Merlon BR (10%)	44,050
Bedaftin 2001 (7.5%)	24,250

N.B.—Tests were carried out on a ring-wear abrasion tester using a No. 1 carborundum stone.

Merlon BR (Monsanto) = Aqueous dispersion of polyvinyl butyral

Bedaftin 2001 (ICI) = "A non-drying oil urea-formaldehyde modified alkyl resin in Cellosolve"

Zinc Chloride versus Urea-Formaldehyde and Melamine-Formaldehyde Resins as a Stiffening Agent for Nylon

To illustrate the permanence of the zinc chloride finish, a comparison was made with the finishes obtained by the use of thermosetting synthetic resins.

Trials were carried out on a twill and a leno fabric and also on a warp-knitted shoe fabric. Samples of these fabrics were stiffened with zinc chloride, with urea-formaldehyde, and with melamine-formaldehyde resins. All the samples were submitted to

TABLE X
Effect of S.D.C. No. 4 Washing Test on Fabrics stiffened with Zinc Chloride and Resins

Fabric	Treatment	Handle and Appearance	
		Before Washing	After Washing
Twill*	5% Zinc chloride soln.	Stiff	Partial breakdown—moderately stiff
	15% Zinc chloride soln.	Very stiff	Almost unaffected
	5% (by wt.) Melamine-formaldehyde resin	Moderately stiff	Almost complete breakdown
Warp-knitted†	5% Zinc chloride soln.	Stiff	Considerable breakdown
	15% Zinc chloride soln.	Very stiff	Almost unaffected
	8-5% (by wt.) Melamine-formaldehyde resin	Stiff, slightly yellow	Almost complete breakdown
	100 parts Urea-formaldehyde cold-cure resin + 40 parts water	Very stiff	Considerable breakdown
Leno‡	5% Zinc chloride soln.	Stiff, poor-moderate resistance to yarn slippage	Considerable breakdown
	15% Zinc chloride soln.	Very stiff, resistant to yarn slippage	Almost unaffected
	100 parts Urea-formaldehyde cold-cure resin + 40 parts water	Very stiff, resistant to yarn slippage	Complete breakdown and destruction of fabric

* 2/1 Twill—
Warp ... 210/34/748 nylon yarn
Weft ... 210/34/8 nylon yarn
Ends per inch ... 77
Picks per inch ... 74

† Constructed from 2-fold/100/34 nylon yarn

‡ Warp ... 2-fold/210/34/58 nylon yarn
Weft ... 2-fold/210/34/58 nylon yarn
Ends per inch ... 56
Picks per inch ... 56
Single-end leno, 2 picks running as one

an S.D.C. No. 4 washing test³, the results being summarized in Table X.

These samples show that the resistance to very severe washing of fabric stiffened with zinc chloride is much better than with melamine- or urea-formaldehyde resins. The reason is probably that resins are essentially surface applications, whereas the zinc chloride treatment involves fusion of the nylon. Breakdown of the zinc chloride finish can be effected only by severe mechanical action, and generally speaking, therefore, the finish is to be regarded as permanent except in cases where the degree of stiffening is only slight. On the other hand, attractive finishes can be obtained by the use of synthetic resins and, provided that an exceptionally high degree of permanence is not required, such resins will be used on nylon.

Thread Slippage Resistance

To ascertain the effect of the treatment upon thread slippage resistance and other properties a trial was carried out on a works scale. Four five-yard lengths of scoured nylon marquisette were treated as follows—

(1) and (2)—Impregnated with 0.25% and 0.5% zinc chloride by weight respectively, dry-heat-set and stiffened, then dyed with disperse dyes on a jig.

(3) and (4)—Dry-heat-set, dyed with disperse dyes, impregnated with 0.5% and 1.0% zinc chloride on weight of fabric respectively, dried and stiffened at 115°C. (239°F.), washed, then treated with soda ash, and dried.

Stiffening comparable with laboratory results was obtained in all cases. Some uneven treatment occurred, mainly owing to the open structure of the fabric and the processing of relatively short lengths of material. However, slight modification of processing conditions could overcome these troubles.

The treated samples were then compared for breaking load-extension and seam slippage, also for wash fastness and dimensional stability, with a steam-set and a dry-heat-set sample which had been subsequently stiffened by a resin finish as a

final operation. The results are recorded in Tables XI and XII.

TABLE XII
Dimensional Stability and Wash Fastness of Stiffened Marquisette Fabric*

Fabric Treatment†	Dimensional Shrinkage (%)			Resistance to S.D.C. No. 2 Washing Test (three washes)‡
	Warp	Weft	Area	
(A) ...	2.5	2.5	5.0	Appreciable loss in stiffness
(B) ...	2.5	2.5	5.0	Appreciable loss in stiffness
(1) ...	2.4	3.3	5.6	Slight loss in stiffness
(2) ...	2.0	2.1	4.6	Slight loss in stiffness
(3) ...	2.1	1.9	4.0	Slight-moderate loss in stiffness
(4) ...	2.7	2.6	5.2	Slight-moderate loss in stiffness

* Fabric construction same as in Table XI

† For details of treatments, see Table XI

‡ All treatments fast to one wash

Effect of Zinc Chloride on the Shade of Dyed Fabrics

Samples of paranylon and 8-oz. nylon canvas were dyed with a wide range of disperse, acid, and chrome dyes. The samples were then stiffened using varying concentrations of zinc chloride.

In general, it appears that the resistance of dyes to this process is good, especially with the faster anionic type. On the other hand, care must be taken in the selection of disperse dyes if shade changes are to be avoided; this is especially so with yellows. However, a shade change on processing does not necessarily preclude the use of a particular dye, because the change in shade can be allowed for provided that the dyer has knowledge of the change to be expected.

Fabric may, in some cases, be dyed after stiffening, but the adoption of this technique and its manipulation will depend upon the stiffness of the fabric and its ease of handling on the machinery available. In all cases, it is important that the impregnation with zinc chloride solution and subsequent heating conditions be uniform for level results to be obtained.

Arising from the work so far carried out, it is apparent that great care must be exercised in the choice of suitable processing conditions. The

TABLE XI
Breaking Load-Extension and Resistance to Slippage of Marquisette Fabric*

Process	Breaking Load (lb.)		Extension at Break (%)		Warp slipping on Weft		Weft slipping on Warp	
	Warp	Weft	Warp	Weft	Resist. (lb.)	Extens. (%)	Resist. (lb.)	Extens. (%)
(A) Control (heat-set and resin-stiffened) ...	69.0	35.2	28.6	27.1	10.9	19.6	36.8	22.4
(B) Control (steam-set and resin-stiffened) ...	70.4	40.6	36.2	33.1	10.0	21.5	34.5	24.8
(1) 0.25% Zinc chloride, dry-heat-set, and stiffened ...	75.5	48.8	41.1	43.3	47.4	54.4	59.7	43.3
(2) 0.5% Zinc chloride, dry-heat-set, and stiffened ...	56.8	50.3	35.5	45.5	47.1	44.4	50.9	35.5
(3) 0.5% Zinc chloride, dried at 115°C., and stiffened ...	50.5	40.8	35.5	36.6	35.0	38.9	62.5	36.7
(4) 1.0% Zinc chloride, dried at 115°C., and stiffened ...	60.4	40.5	36.7	37.8	24.0	41.1	62.6	41.1

* FABRIC CONSTRUCTION—

Warp ...	45/15/308 nylon yarn
Weft ...	45/15/308 nylon yarn
Knots per inch ...	78
Picks per inch ...	55
Weave ...	Single-end leno

degree of stiffness for any one fabric is dependent upon concentration of zinc chloride and temperature at which the treated fabric is heated. By control of these two variables it is possible to obtain a range of stiff finishes without incurring excessive yarn degradation. It has been shown that for any given concentration of zinc chloride the final degree of stiffness obtained is related to the temperature of heating, the stiffness increasing progressively with temperature. This is thought to be due to the controlled liberation, *in situ*, of hydrochloric acid, which causes fusing to take place; the higher the temperature, the greater the amount of hydrochloric acid liberated. Allied to this is the increased activity of the acid with increase in temperature and the possibility of the zinc itself playing some specific part in promoting the reaction. If the concentration of zinc chloride and final temperature of heating are too high, dissolution of the nylon occurs. The limitations of the process on lightweight nylon fabrics must be clearly recognised. It is also important that faulty application of the process cannot be rectified and, therefore, precise operating conditions must be determined by experiment before proceeding to bulk runs.

It is recommended that the stiffened fabrics be well washed after final heating to remove undecomposed zinc chloride, so that neutralisation in mild alkali solution does not cause deposition of zinc hydroxide. This is more essential when the stiffening has been developed at low temperatures.

Some of the uses to which a zinc chloride finish on nylon may be applied are—

- (a) Prevention of thread slippage (e.g. on open leno and certain voile-type fabrics)
- (b) To increase abrasion resistance and reduce filamentation (e.g. on shirt collars made from nylon warp-knitted fabrics)
- (c) For stiffening collars, cuffs, and fronts of nylon shirts, interlining fabrics, and fabrics for inclusion in foundation garments
- (d) For imparting "firmness" to lightweight fabrics such as marisettes, lace, and net
- (e) For use on heavy warp-knitted or woven fabrics for shoe uppers, travel bags, etc.

The authors wish to thank British Nylon Spinners Ltd. for permission to publish this paper.

BRITISH NYLON SPINNERS LTD.
PONTYPOOL
MONMOUTHSHIRE

References

- ¹ Mecklenburgh, G. K., *J. Textile Inst.*, **41**, p 161-P 175 (1950).
- ² *Second Report of the Fastness Tests Committee* (2nd edition, Bradford 1950. Society of Dyers and Colourists), p. 5.

Discussion

Mr. A. S. CLULEY: It is well known that the use of "non-slip" resin treatments on woven fabrics decreases the tear resistance. Is a similar effect produced by the zinc chloride treatment?

Mr. MECKLENBURGH: In common with resin treatments used to give stiffened or non-slip finishes on woven fabric, the zinc chloride treatment pro-

duces a reduction in tear strength. If the process is applied correctly to avoid chemical degradation, then the reduction is due largely to the mechanical effect, as is the case with resin treatments.

Mr. H. W. PARTRIDGE: Has the lecturer or his colleagues done any work on the effect of varying conditions of steam setting on dyeing properties?

Mr. MECKLENBURGH: In general, the rate of dyeing on steam-set fabric is increased over that on unset fabric. The sensitivity of dyes to setting conditions varies with those setting conditions and from class to class; some anionic dyes are very sensitive to changes in orientation of the molecular chains, and show increased rate of dyeing on pressure-steam-set fabric, whereas disperse dyes are relatively insensitive. It is important, however, that setting conditions be uniform in order to minimise dyeing irregularities.

Mr. J. H. BASS: What is the nature of the delustring action of zinc chloride? Can the lecturer give any explanation of the occurrence of a white deposit in the fabric when dyed goods are so treated?

Mr. MECKLENBURGH: The delustring action of the zinc chloride process may be due to either—

- (a) Pitting of the surface of the nylon yarn causing light scattering, or
- (b) Presence of zinc oxide embedded in the surface layers of the yarn.

A white deposit on dyed goods may be obtained during neutralisation with alkali if unreacted zinc chloride or oxychloride is present on the fabric. A weakly acidic wash will remove this unreacted zinc chloride and thus prevent the deposition on the goods of zinc hydroxide or oxide during neutralisation.

Mr. GIBSON: Do the figures given refer to the amount retained by the nylon, or to the actual strength of the padding solution? What would be the optimum expression on squeezing?

Mr. MECKLENBURGH: The figures are quoted as solution strengths except in Tables XI and XII, where they are quoted as percentage weights on fabric calculated from solution strength and mangle expression. On continuous-filament nylon fabric, the optimum expression is about 50%; higher expressions such as 80% would introduce liquor migration troubles during subsequent processing.

Mr. S. A. WELCH: Can information be given on the precise nature of the chemical reactions involved in the zinc chloride process?

Mr. MECKLENBURGH: It is suggested that the stiffening obtained with the zinc chloride process is due to the controlled liberation, *in situ*, of hydrochloric acid during baking, which causes dissolution of the nylon.

Mr. J. RAYMENT: Not all the zinc salts appear to be removed when the set fabric is rinsed. Is any information available on the amount of zinc retained by the nylon, and in what form?

Mr. MECKLENBURGH: After rinsing and neutralising, the yarn will probably contain zinc oxide. The maximum amount retained can be calculated from the processing conditions; the precise amount retained would necessitate chemical analysis.

PUBLICATIONS SPONSORED BY THE SOCIETY'S FASTNESS TESTS
CO-ORDINATING COMMITTEE—IX

Investigations into Fastness to Perspiration

PERSPIRATION FASTNESS SUBCOMMITTEE

Until comparatively recent times tests employed in assessing fastness to perspiration have been of an unsatisfactory nature. Thus, in the first edition of the *Colour Index*, published in 1924, no test is given for wool, and for cotton there is only a somewhat indefinite test using a solution of acetic acid.

In 1934 the Society published its *Report on the work of the Fastness Committee in fixing Standards for Light, Perspiration and Washing*, in which a detailed account is given of the constitution and reactions of natural perspiration. It was found that, in the large majority of cases, freshly shed perspiration possesses an acid reaction, and gradually becomes alkaline owing to bacterial action. As a result of this investigation, the Society put forward two artificial perspiration liquors, one at pH 5.6 and the other at pH 7.8, and devised a method for their use called the "plate method". Since that time much work has been done, and this has confirmed the suitability of the plate method for this type of test.

At the same time other countries carried out work on this subject and introduced artificial tests, one being that of the Deutsche Echtheitskommission. This uses one solution, containing salt and ammonia at pH 11.1, in which the tests are carried out for 30 min., following which the liquor is made acid by an addition of acetic acid to pH 4.7, and the test continued for a further 30 min. This test was the standard German test up to the outbreak of war in 1939.

At the present time there are three main groups concerned with fastness testing, viz. the Continental (E.C.E.), American (A.A.T.C.C.), and British (S.D.C.), and all have put forward tests for the assessment of perspiration fastness.

Following the meeting held at Buxton in June 1948 under the auspices of the International Standards Organisation, at which fastness testing was discussed, the Fastness Tests Co-ordinating Committee set up a subcommittee to study these various perspiration tests, and any others which might be in existence, with the object of arriving at a test which might command international agreement.

Examination of available information indicated that, although several countries have official methods for testing fastness, in each case this is either identical with, or a modification of, a test put forward by one of the main groups mentioned above, or is of such a nature as to be of little value for the purposes of reasonably accurate assessment. It was evident that the three main proposals were the only ones which required consideration.

CONTINENTAL METHOD

This test is a slight modification of the German test, given above, in that the solution used contains common salt and disodium phosphate; treatment

is carried out at pH 9.5 for 30 min. at 45°C., the liquor is then acidified to pH 4.7 with acetic acid, and treatment is continued for a further 30 min.

It seems somewhat strange that, while it is known that natural perspiration gradually changes from a pH below to one above 7, this test should be carried out in the reverse order; as far as is known, there is no reason for this choice of procedure. Apart from this, the pH range appears to be wider than would be experienced in practice, and the temperature is higher than the temperature of the human body. In addition, it has been found that the test is not easy to apply in view of the necessity for repeated adjustment of pH, and a further point is that the liquor ratio is widely removed from that which obtains under conditions of actual wear. The Subcommittee were unanimous in the opinion that a simpler method should be recommended, and one much more in conformity with wearing conditions.

AMERICAN METHOD

This method involves the use of two solutions, acid and alkaline respectively, the original procedure being to incubate patterns, thoroughly wetted with these solutions, in tubes at 100°F. for four periods ranging from 40 min. to 18 hr.

A variation of this method, given as a tentative test, is to use a mechanical device called a *Perspirometer*, in which the patterns are clamped between glass plates, after saturation with the solution, under a pressure of 10 lb., and the whole pack is incubated at 100°F. for 6 hr.

It is interesting to note that both the solutions used are buffered with disodium phosphate, although not sufficiently strongly to deal with all possible variations in the pH of dyed wool patterns.

Tests of the Perspirometer method in comparison with the S.D.C. plate method revealed no marked difference in results, using identical solutions, except that the latter was rather more severe as regards bleeding. This is probably due to the patterns in the Perspirometer requiring a somewhat longer time to attain the specified temperature and also to the smaller amount of liquor they contain. Although this difference is not of great moment, it is considered that the plate method is much to be preferred on the grounds of simplicity and availability of the necessary apparatus.

BRITISH METHOD

This method, published in the *Second Report of the Fastness Tests Committee*, uses one solution only, viz. 3 g. sodium chloride per litre adjusted to pH 7.8 by means of sodium bicarbonate, and the patterns, after preliminary neutralisation in the case of wool and silk, are treated by the plate method at 37°C. for 4 hr. In putting forward this

test it was considered that both an acid and an alkaline test were unnecessary, as—

- (1) In the case of animal fibres an alkaline test is in general more severe.
- (2) In the case of vegetable fibres a water test in any event is more severe than a test using a solution containing salt. As is well known, natural perspiration contains salt, the concentration being approximately that used in the S.D.C. test.

Such was the position when the Subcommittee was formed to re-examine the whole matter.

NECESSITY FOR BOTH ACID AND ALKALINE TESTS

The first point considered was the difference which could be produced by using both acid and alkaline tests, and an examination was made with various types of dyes. As expected, it was found that with wool dyes in general an alkaline test is more severe, and with basic dyes the reverse is the case. Neolan-type dyes gave a slightly worse result acid than alkaline, while direct and disperse dyes appeared to give similar results with both. The only case where a marked difference in favour of an acid test was noted was thus with basic dyes, and on the whole it did not appear possible to make out a very good case for the use of two separate test solutions.

There was, with certain dyes, a small change in colour, but this is of much less importance than similar changes which are referred to below.

CONSTITUTION OF TEST LIQUOR

The Subcommittee quickly came to the conclusion that no artificial testing solution, so far suggested, imitated the effect of natural perspiration on a number of dyeings, the best known example being Solway Blue BS (C.I. 1054). If this had been an isolated case it would have been of minor importance, as a similar result can be obtained by using a more concentrated salt solution, e.g. 20 g./litre, although even then the shade change produced is not exactly like that with natural perspiration. More important observations related to dyeings on cotton made with direct dyes which either contain copper in their molecule or are subjected to an aftercoppering treatment subsequent to dyeing. Thus the shade of a dyeing of Solar Red Brown 2RL (8), a copper-containing dye, changes from red to orange when spotted with natural perspiration and then allowed to dry, and this change is enhanced by repeated treatment. Similar changes were observed with dyes whose fastness properties are improved by aftercoppering, e.g. Chloramine Sky Blue FF (C.I. 518) and Coprantine Orange 2BRL (Ciba). The change in shade is in most cases accompanied by a decrease in the fastness to light, and analysis reveals that it is due to removal of copper from the dye complex. Treatment of the perspiration-treated samples with copper salts results in restoration of the original properties of the dyeing.

One further observation of importance has also been made—When a pattern of cotton fabric, which had been worn so as to be subjected to the action of natural perspiration, was placed in loose

contact with a dyeing of Coprantine Orange 2BRL, the latter dyeing was discoloured in a similar way to the discoloration caused by the direct action of perspiration. This effect appears to be the result of the presence of some volatile constituent in natural perspiration.

CONSTITUENTS OF NATURAL PERSPIRATION

The results obtained by Mosher from the analysis of six samples of perspiration taken from three subjects are given in the *Report of the Society of Dyers and Colourists' Fastness Committee* (1934).

Members of the present Subcommittee have carried out tests with several substances reported by Mosher to be constituents of natural perspiration. It was found that a solution containing approx. 30 mg. amino acid nitrogen per litre (glycine being used as the source thereof) produced a change in the shade of copper-containing direct dyes, similar to that obtained with natural perspiration. Further tests were also carried out with certain sulphur-containing substances, viz. thioglycollic acid, thiourea, and cysteine. When solutions of equal strength were spotted on dyeings of Coprantine Orange 2BRL and Solar Red Brown 2RL, the three sulphur-containing compounds were found to be much more effective than glycine in changing the shade of the dyeing.

Table I gives the mean values for the amino acid constituents of human perspiration, according to Heir, Cornbleet, and Bergeim¹, and indicates an amino nitrogen content of 54 mg./litre, which is rather higher than that previously recorded by Mosher. It is to be noted that there is no indication of the presence of any sulphur-containing acid.

TABLE I
Amino Acids in Human Perspiration

Amino Acid	Concn. of Amino Acid (mg./litre)	Concn. of Amino Nitrogen (mg./litre)
Arginine ...	135.8 ± 39.2	21.9
Histidine ...	80.2 ± 9.7	7.2
isoLeucine ...	22.7 ± 6.6	2.5
Leucine ...	26.9 ± 7.7	2.9
Lysine ...	22.6 ± 4.5	4.4
Phenylalanine ...	21.9 ± 6.3	1.9
Threonine ...	53.8 ± 18.4	6.3
Tryptophan ...	11.2 ± 3.3	0.8
Tyrosine ...	31.5 ± 9.5	2.5
Valine ...	29.6 ± 7.5	3.6
		54.0

EXPERIMENTAL

Preliminary tests were carried out with a single solution containing the amino acids in the concentrations given in Table I. Two drops of this solution were spotted together on to dyeings of Coprantine Orange 2BRL and Solar Red Brown 2RL, which were then dried at 40°C. A shade change, similar to that produced by natural perspiration, was produced on the Coprantine Orange 2RL dyeing, but only slight, if any, change could be seen in the Solar Red Brown 2RL dyeing. Repeated treatment, however, produced a change of shade, and after ten treatments (20 drops in all) a very pronounced change had occurred.

Spotting tests on the same dyeings were then carried out using unbuffered solutions of the individual amino acids (a) at a strength of 0.5 g. per litre, (b) at the concentration at which they were reported to occur in natural perspiration. These tests indicated that the amino acid with the most pronounced effect on the shades of these dyeings was histidine.

Further spotting tests were then carried out using (a) solutions of histidine buffered to pH 3, 5, 7, and 9 with a universal buffer mixture containing phosphoric, acetic, and boric acids (Britton and Robinson²), (b) solutions containing the buffer mixture only. It was found that, at pH 3 and 5, the effect of the solution containing the histidine on the shade of the dyeings was little, if any, greater than the effect of the buffer solution alone. At pH 7 and 9, however, the solution containing histidine produced pronounced shade changes which were not given by the buffer solutions alone.

Variation of the histidine concentration of solutions buffered at pH 7 showed that there was a marked increase in its effect on the shade of the Coprantine Orange 2BRL dyeing with an increase in concentration from 0.5 to 1.0 g./litre.

Spotting tests were then carried out using solutions containing 1 g. of the individual amino acids per litre, all the solutions being buffered to pH 7. A single treatment with two drops of each of the amino acid solutions produced a marked change in the shade of the Coprantine Orange 2BRL dyeing, but the solution containing histidine had a more marked effect than any of the others. Only the solution containing histidine produced any appreciable change in shade when the Solar Red Brown 2RL dyeing was similarly treated. It should be noted that the shade changes produced on these two dyeings with the solutions containing 1 g. histidine per litre were obtained by a single spotting treatment, thus giving an effect similar to that obtained with natural perspiration.

These tests show conclusively that, of the amino acids reported as being present in human perspiration,

histidine is the most important as a cause of the alteration in shade of copper-containing direct dyeings. In this connection it is of interest to note that Maley and Mellor³ have determined the overall stability constants for bivalent metal complexes of α -amino acids in water at 25°C. Their results indicate that the stability of the copper complex of histidine is much greater than that of the copper complexes of other amino acids. It is suggested that this stability arises because histidine is attached to a metal atom by groups different from those involved in the co-ordination of the other amino acids. The latter are attached through carboxyl and amino groups, the former probably through amino and imino groups.

CONCLUSION

It seems evident that the effect of natural perspiration on certain dyeings, notably those containing copper, is due to its amino acid content, and of all the amino acids known to be present histidine is by far the most reactive. The Subcommittee believes that, on the basis of the work detailed above, it should be fairly easy to devise an artificial testing solution which will much more closely approximate to natural perspiration in its effect on dyeings than any so far suggested. Work is proceeding to this end, mainly on a wider range of dyes, applied under differing conditions, and it is hoped to publish the results at an early date.

* * *

The Subcommittee wish to acknowledge help in tracing references given by the London School of Hygiene and Tropical Medicine, the British Leather Manufacturers' Research Association, the Medical Research Climatic Unit, Oxford, and the School of Medicine, Manchester University.

(Received 7th August 1952)

References

- ¹ Heir, Cornbleet, and Bergeim, *J. Biol. Chem.*, **166**, 327 (1946).
- ² Britton, *Hydrogen Ions*, **1**, 313 (London: Chapman & Hall).
- ³ Maley and Mellor, *Nature*, **165**, 453 (1950).

CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

Quantitative Relationship between Depth of Dyeing and Light Fastness

It is well known that the light fastness of a dye generally increases with an increase in the depth of the dyeing, but, as far as we are aware, the exact quantitative relationship between depth and fastness has not previously been determined. We have now found that the published information on the light fastness of commercial dyes can be used to determine such a quantitative relationship of apparently quite general applicability.

The normal methods of light fastness evaluation are subject to many random errors, caused not only by the difficulty of judging visually the extent of fading, but also by a variety of chemical and physical influences, e.g. changes in the characteristics of the light source, differences in spectral absorption between dyes, and variations in

atmospheric conditions. If a sufficiently large number of light fastness observations on dyeings of a range of depths are available, however, averaging them will greatly reduce the random disturbances and so will allow an examination to be made of any systematic influences of dye concentration upon fastness.

The light fastness gradings of large numbers of dyes on the principal fibres, determined by means of either the British or the German standards^{*}, are available in commercial pattern cards; and in most cases data are given for three depths of each dye. In Fig. 1 are shown the results of averaging these mean light fastness data for particular classes of dye, and it is seen that there is a straight-line relationship between the fastness grade and the

* The two series of standards are very closely similar, indeed the patterns representing several grades are identical in both¹.

logarithm of the percentage depth. We have now shown that the reaction order of the light fading process may be calculated from the slope of the curves, and it is found to lie between zero and unity. A likely interpretation of this fact is that the fading process takes place at the interface between the atmosphere and the submicroscopic aggregates of the dye on the fibre.

When account is taken of the number of determinations, or rather of the degrees of freedom, the gradients of these lines differ significantly from one another. It is thus possible to interpret the slope of each curve, and hence the reaction order, as being dependent on some property or properties of the respective classes of dye, of which the degree of aggregation of the colouring matter in the fibre may well be the most important. It will be observed that there is a tendency in certain cases for the slope to increase with increase in the expected degree of aggregation. Thus, in the examples illustrated in Fig. 1, it is seen that the slope increases in the order—direct cotton < vat < azoic dyes.

An interesting fact is that dyes of the same class, but made by different manufacturers, in some cases give different slopes. The following reasons for this may be suggested—

(a) Differences in the extent of fading customarily allowed in assessing fastness.

(b) Differences between the average humidity of the atmosphere where the determinations were made, which might affect the aggregation of the dyes in the fibre.

(c) Variation in the manufacturers' criteria, other than light fastness, which determine the suitability of a dye for sale, since the general properties, including light fastness, of a class of dyes will, of course, depend on the method of their selection.

It should be pointed out that the percentage figures used to determine the data of Fig. 1 refer to the amount of dye present in the dyebath at the start of dyeing and not to the amount actually present on the fibre*. The quantities of dye involved are in each case much less than the maximum capable of being absorbed by the fibre, so that the figures given should, by the laws of absorption, be closely proportional to the amount actually taken up, and the use of the depths in the way described is thus believed to be justified.

Some interesting corollaries follow from a study of the facts revealed in Fig. 1. Amongst these are—

(1) If the light fastness of a dye on a given fibre can be determined with confidence, the specification of its value at two depths will be sufficient for the prediction of the value at any other depth, by interpolation on the appropriate curve. This fact should not only be of obvious value to all dye users, but it should also assist dye manufacturers by simplifying the routine testing of light fastness of new products.

(2) Azoic dyes, especially, tend to have curves with a greater slope than the water-soluble direct dyes. In other words, these insoluble dyes show a

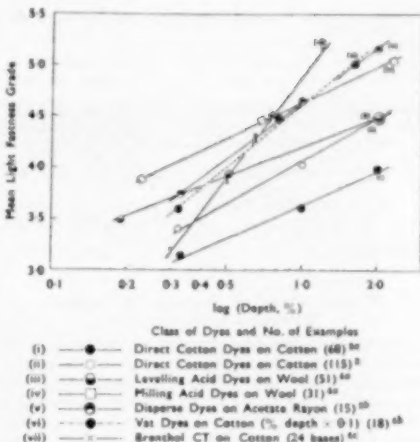


FIG. 1.—Relationship between Mean Light Fastness and Logarithm of Depth (%).

relatively greater improvement in light fastness as the depth is increased than do the soluble ones. The reason is probably that the insoluble dyes are present inside the fibre in larger aggregates than are the soluble ones. This fact may have an important practical implication, for high light fastness in medium or deep shades may perhaps be more likely to be attained with dyes which can be changed into an insoluble form on the fibre than with those which remain soluble after application.

Ricketts⁵ has shown that the British standards are evenly spaced in geometrical order between numbers 1 and 6 inclusive. In practice, however, patterns lower in fastness than Standard 1 may be specified as of fastness grade 1, so that in compiling the data for Fig. 1, we have included only those dyes in which all the dyeings given have light fastness numbers between 2 and 6*, the intermediate grades 2-3, 3-4, 4-5, 5-6 being calculated as 2.5, 3.5, 4.5, 5.5 respectively. The data for vat dyes are included for interest, but they are not strictly representative of this class of dye, because only a selected few from the whole range fall within the required fastness limits.

Further details of this work will be published elsewhere.

* * *

We wish to thank the Geigy Co. Ltd., Imperial Chemical Industries Ltd. (Dyestuffs Division), and Sandoz Ltd. for the gift of the publications used in this work.

J. C. EATON

DEPARTMENT OF MATHEMATICS

C. H. GILES

MANFRED GORDON

DEPARTMENT OF TECHNICAL CHEMISTRY

ROYAL TECHNICAL COLLEGE
GLASGOW C.1

22nd July 1952

* The data for the Brenthol series do actually refer to the percentage of Brenthol present on the fibre.

* Three figures below 2 are included in the Brenthol CT data.

¹ Fastness Tests Co-ordinating Committee, J.S.D.C., 67, 188 (1951).

² Geigy Co. Ltd.—

(a) *Direct Dyestuffs for Cotton*, No. 510 (1951);

(b) *Schlagl Direct Dyes for Acetate Rayon*, No. 507 (no date).

³ Sandoz Ltd., *Direct Colours on Cotton*, No. 901 (no date).

⁴ Imperial Chemical Industries Ltd., Dyestuffs Division—

(a) *Acid Dyestuffs on Wool*, Code No. 550/E(H)/212/8, 50;

(b) *Caledon and Dacron Direct Dyestuffs for Cotton*, Code No. 548/E(h)/212/950;

(c) *Brenthol-dyed Materials. Fastness to Light in Deep to Pale Shades* (Brenthol Pamphlet No. 4), Code No. 114/112/237 (no date).

⁵ Ricketts, R. H., J.S.D.C., 65, 200 (June 1952).

Measurement of Damage in Coarse Keratinous Materials

Although a great deal of attention has been given to the assessment of damage in wool materials, comparatively little work has been done on damaged coarser fibres such as bristle, horsehair, and squirrel hair. These fibres are not normally used for textile purposes, but find their main application as brush-filling materials or as upholstery fillings.

A study of the effects of chemical and mechanical treatments on the coarser keratinous materials has shown that methods such as alkali solubility are not sufficiently sensitive for use with these materials. Moreover, often only short lengths of material are available, and it is not possible to determine load-extension characteristics. Microscopic appearance is valuable if the damage is severe, but staining tests have only a limited application because frequently the coarser fibres are black or heavily pigmented. Because of the limitations of the well established tests, the possibility of using the resistance to bending as an indication of damage was considered, since this characteristic is clearly of importance in the performance in practice of the materials used, and moreover, since they are comparatively thick, the measurement of bending does not present any particular difficulty. The method finally adopted was to measure the percentage increase in the deflection of the free end of the fibre on wetting (I.D.W.), since this was found to be almost independent of the dimensions and shape of the cross-section of the fibre, whereas the actual deflection is greatly dependent on these. To obtain precise values some twenty determinations should be made, but in practice it is possible to obtain valuable information from as few as five determinations.

It has been found that sound materials give values between 30 and 70% for the I.D.W., while damaged materials give higher values depending on the degree of damage. The method therefore becomes a useful sorting test even if only a few fibres are available. Brief details of the method employed are as follows, and some typical results are given in Table I.

When selecting a fibre for test the tip is discarded and the fibre is freed from extraneous

matter. The fibre is immersed in water for 24 hr., and then gripped lightly near one end so that a 2.5 cm. length projects horizontally. Shorter lengths may be tested if desired. The grip and fibre are immersed in water in a tank with plate glass sides. A glass rider (0.003–0.172 g.), sufficient to give a deflection of 0.1–0.5 cm., is placed on the free end of the fibre, and the deflection is measured. The fibre is allowed to dry, it is again gripped, and the deflection of the dry fibre under the same load is measured. To correct for the upthrust of the water on the glass rider, the deflection of the wet fibre is multiplied by 1.66, the value for the I.D.W. % being given by the expression—

$$\frac{(\text{Deflection wet} \times 1.66 - \text{Deflection dry})}{\text{Deflection dry}} \times 100$$

All the measurements are carried out in a room maintained at 65% R.H. and 22.2°C.

TABLE I

	BRISTLES	I.D.W. (%)
4½ in. White Gostorg	...	40
3 in. Black Chungking	...	40
3½ in. White Polish	...	50
6 in. Black Indian	...	60
85 mm. German	...	60
	HAIRS	
Human	...	60
Cow tail	...	70
Skunk	...	60
Ox ear	...	50
Badger	...	30
Squirrel	...	50
Ringcat	...	40
Civet	...	50
Horse mane	...	60
Horse tail	...	40
Kolinsky	...	50
Weasel	...	40
	DAMAGED BRISTLES	
3½ in. Black Chungking bristle after boiling for 6 hr.	...	80
2½ in. Unbleached white Chungking bristle after sulphur stoving and boiling for 2 hr.	...	100
3 in. Black Chungking bristle after steaming for 4 hr. at 20 lb./sq. in.	...	140
White Japanese bristle damaged during bleaching	...	180
White Chungking bristle, from brush, damaged by alkali	...	220
Black bristle, from brush, damaged by acid	...	280

The authors wish to express their thanks to the Council of the British Brush Manufacturers' Research Association Ltd. for permission to publish an account of this work.

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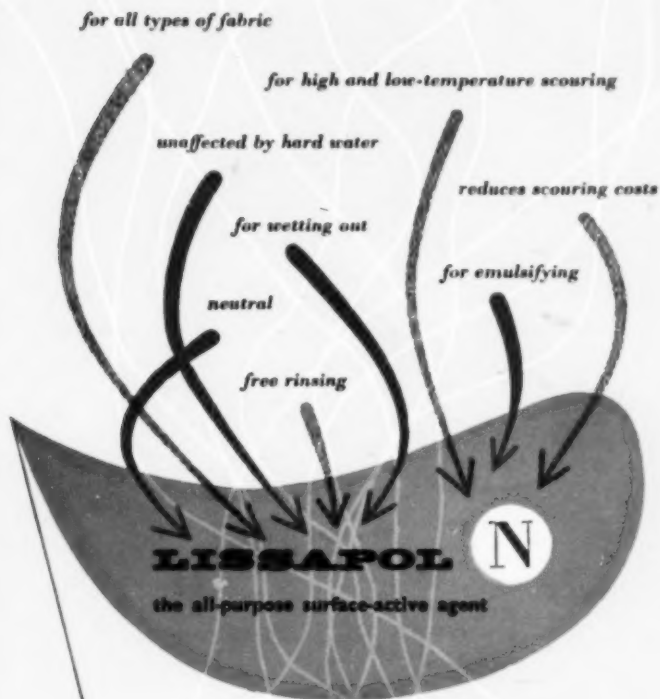
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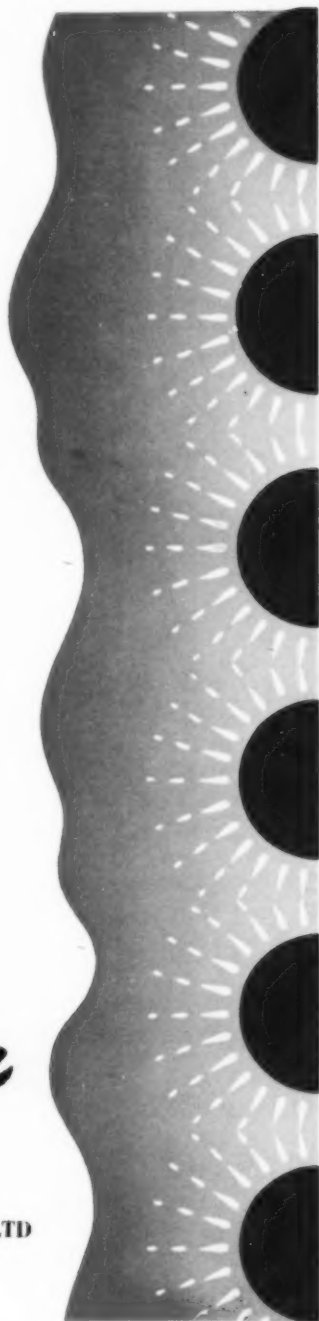


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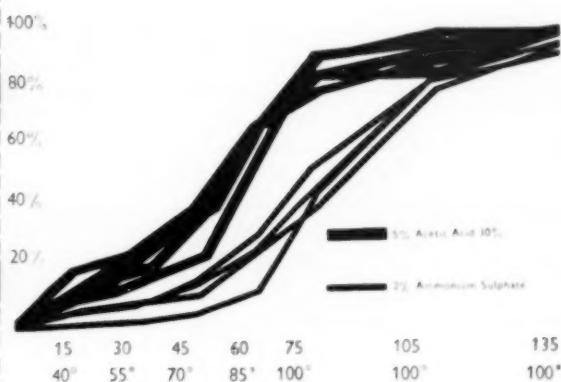
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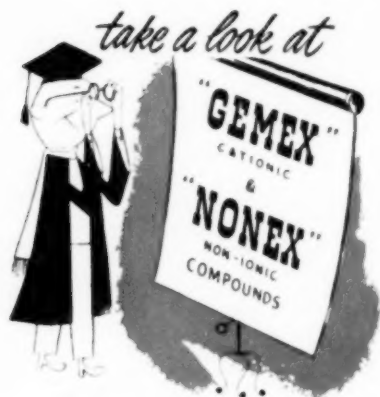
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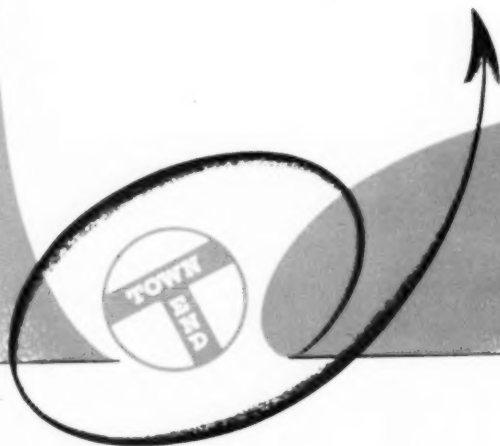
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ERRATA

New Data on the Chemical Composition of Oxycelluloses. E. D. Kaverneva (*J.S.D.C.*, 67, 397 (Oct. 1951)); **Chemical Changes produced in the Cellulose Macromolecule by Oxidising Agents.** E. D. Kaverneva *et al.* (*J.S.D.C.*, 68, 329 (Aug. 1952))—The results said to be obtained by the "phosphomolybdate" reaction were in fact obtained by the **phosphotungstate** reaction.

The Reduction Properties of Vat Dyes. W. J. Marshall and R. H. Peters (*J.S.D.C.*, 68, 298 (Aug. 1952))—In references (2) and (4), "Rogers" and "Clarke" should be **Royer and Clark** respectively.

Recent Developments in Bleaching with Hydrogen Peroxide. W. S. Wood and K. W. Richmond (*J.S.D.C.*, 68, 337 (Sept. 1952))—The name of the chairman should be **Mr. H. A. Brassard**.

Notes

Meetings of Council and Committees September

Council—No meeting
Publications—16th
Colour Index Editorial Panel—25th
Fastness Tests Co-ordinating—18th
Terms and Definitions—3rd
Examinations Subcommittee—1st
Review of Textile Progress—29th

New Dyestuffs Advisory Committee

The Board of Trade announces that the members of the new committee are—

<i>Chairman</i>	Sir William Palmer, K.B.E., C.B.
<i>Deputy Chairman</i> ...	Mr. E. J. Holford-Strevens
<i>Department of</i>	
<i>Government Chemist</i>	Mr. B. A. Ellis
<i>Association of British</i>	Mr. H. Jackson
<i>Chemical Manufacturers</i>	Mr. H. Blackburn
<i>(Group D)</i>	Mr. H. Boothroyd
<i>Colour Users' Association</i>	Mr. N. G. McCulloch, C.B.E.
	Mr. D. Carter
	Mr. A. R. Edge
	Mr. J. G. Evans
<i>British Colour Makers' Association</i>	Mr. H. Gosling
<i>Secretary</i>	Mr. C. G. Hulse, Dyestuffs Office

β -naphthol, 3-amino-1:5-naphthalenedisulphonic acid, phthalic anhydride, anthraquinone, and quinoline. c o c

du Pont's 150th Anniversary

On 18th July 1952 E. I. du Pont de Nemours & Co. celebrated the 150th anniversary of its founding by Eleuthère Irénée du Pont. For the first century of its existence the firm was concerned solely with making explosives, but a decision by the U.S. Federal court in 1911 resulted in the business being split into three firms—du Pont, Hercules Powder Co., and Atlas Powder Co. This decision led the du Pont family, who retained control only of the company bearing their name, to start research in other fields, commencing in that of artificial fibres and lacquers from cellulose. The first World War resulted in the company starting to make dyes, but it was not until the 1930s that the dyemaking venture began to pay back its initial cost. This did not deter the company from large-scale expenditure on fundamental basic research, and as a result the company was able in October 1938 to announce the introduction of nylon. During the greater part of the vast expansion of the company's activities and interests during the last half-century, it was under the successive leadership of three brothers—Pierre Samuel, Irénée, and Lammot du Pont—great-grandsons of the founder of the firm. c o c

Export of Dyes Freed

An Open General Licence, issued by the Board of Trade and operative from 1st September 1952, permits the export of synthetic organic dyes, with some exceptions, to all destinations except China, Hong Kong, and Tibet.

Imports of Coal-tar Products and Dyes into the U.S.A. in 1951

In 1951 imports of finished coal-tar products were (all figures being millions) 5.2 lb. worth \$7.3 compared with 5.7 lb. worth \$5.3 in 1950. Of these, dyes amounted to 4.2 lb. worth \$6.3, and for the first time since 1939 Germany was the principal supplier, Swiss imports being the second largest. Imports of coal-tar intermediates were 8.5 lb. worth \$2.2 (1950 figures being 5.5 lb. worth \$1.6), and came mainly from Germany and the U.K. Naphthalene, aniline, and o-cresol were the three chief intermediates imported, but there were substantial imports of p-chloronitrobenzene,

German Export of Dyes in 1951

German exports of dyes in 1951 were worth approx. \$150,000,000 out of a total of \$500,000,000 for all chemical exports. In 1938 Germany produced about one-quarter of the world's production of chemicals, but now she produces only 6%. The drop in her share is partly due to increased production in other countries, the U.S.A. now producing one-half of the world's total production. c o c

Japanese Production of Dyes in 1951-1952

In the year ending 31st March 1952 the annual Japanese production of dyes increased by 2300 tons to a total of 14,720 metric tons. Exports during the year were 899 tons, a decrease of 1466 tons. c o c

Redon Polyacrylonitrile Fibre

Redon is a polyacrylonitrile fibre now being produced by dry spinning by the Phrix-Werke A.G. of Hamburg. c o c

New Books and Publications

An Introduction to Modern Textile Printing
Pp. 74. Paper.

The Printing of Textile Fabrics by Block and Screen

A Practical Manual for use in Colleges and Schools of Art

Third edition. Pp. 68. Paper.

Manchester: Imperial Chemical Industries Ltd., Dyestuffs Division. No price.

Textile printing is one branch only, although an important and highly developed one, in the complete range of processes by which coloured textiles are produced. It is easy, therefore, to understand why textbooks on printing are not numerous. The comprehensive ones are necessarily large and expensive, because they must contain the complicated and detailed information required by the colourist. Even with the best, the beginner may find it difficult to get a clear picture of the operations of the industry and the commonsense reasons behind them.

The first of the present books does not attempt the impossible feat of turning the lay reader into a skilled printer. Nor is it, primarily, a condensed book of reference for the experienced printer. From a wide and confident knowledge, the materials and styles, the operations and machinery are explained and described in simple and easily understood terms. Block, screen, and machine printing are covered, and, in addition, something is said of those properties of textile materials which chiefly interest the printer. Outlines are also given of the preparative processes and the finishing of the cloth after printing. It is surprising how so much has been included without making the book difficult to read. An extremely useful feature is the large number of technical terms which have been defined and explained. In the foreword is an apology for lack of illustration. The difficulties mentioned there will be fully appreciated, but it is certainly true that a few line diagrams to illustrate printing operations would have been very useful.

For students of textile processing, the operations of printing can be utilised very effectively as practical exercises. In such exercises it is possible to illustrate the chief technical properties of dyes very graphically. At the same time, a useful discipline in manipulation is afforded, for the careful worker is rewarded with a result in which his care is made immediately obvious. In another direction, students of design were not well placed, before the present wide industrial use of the silk screen process, for reproducing with their own hands the designs they had made, making use of processes actually current in the industry. Even now, probably as a continuing effect of this former disability, undue emphasis tends still to be placed on woven design both in instruction and in competition work.

The second of these little books represents a skilful and painstaking effort to bring together in a compact form the information that is necessary for successful practical printmaking. Again, it is sur-

prising how much cogent advice and material have been packed into so small a compass. Special attention has been paid to the important details of screen making, and difficulties that may confront the beginner in the preparation of thickenings and colour pastes are smoothed out. An attempt has been made to cover a fully representative range of printing styles on fabrics of all the usual natural and manufactured fibres without an undue multiplication of recipes. Advice on simple steaming arrangements is given, and there are interesting sections on batik and pigment printing. One chapter, not often well done in the *trade-mecum* of this kind, shows how to identify the textile fibres in the fabric to be printed. Of particular value, especially to the non-technical reader, is a long list of firms in this country who can supply the necessary working equipment, materials, and chemicals.

With these generous contents, it may seem ungracious to ask for more, but it may be felt that, while the technical teacher and student can find here all that they could possibly want, those interested purely in design may at first be a little embarrassed by the large number of dyes that can be applied, and the multiplicity of printing styles available. Since a wide variety of viscose rayon fabrics can now be obtained, bright and matt, spun and continuous, might not this be taken as a standard textile material for trials, and a simple palette chosen from easily applied, bright dyes like the Soledons, perhaps with a reinforcement from the Brentogens for bright reds? This would meet many of the demands of the design student until confidence were obtained. Then the more ambitious could adventure among the other styles.

H. A. TURNER

Annual Reports on the Progress of Chemistry for 1951

Vol. XLVIII. Pp. 429. London: The Chemical Society. 1952. Price, 25s. 0d.

This year's Report represents the final stage in implementing the changeover to true Annual Report form, and it is thought that the present framework will prove satisfactory in the future. It contains six chapters together with author and subject indexes and a list of the principal periodicals consulted.

Chapter I deals with *General and Physical Chemistry* in five sections—the quantum theory of valency, molecular structure, the mechanism of chemical change, the statistical theory of solutions, and colloids and surface chemistry. Emphasis is laid in these sections on recent progress in the more fundamental theory of chemical bonding, on microwave spectra, on recent work on non-electrolyte solutions in which the molecules are not too disparate in size, and on coagulation, surface activity, bulk properties of micellar colloids, and emulsions.

Chapter II is on *Inorganic Chemistry*.

Chapter III, on *Organic Chemistry*, occupies nearly one-third of the volume, and has eight large

sections, devoted to theoretical organic chemistry, stereochemistry, general methods, aliphatic compounds, sugars, homocyclic compounds, heterocyclic compounds, and macromolecules. On the theoretical side, considerable progress has been made in applying quantum theory to the prediction of chemical reactivity, and a noteworthy feature is the postulation of ionic intermediates of non-classical structure in organic reactions; e.g. in electrophilic addition to olefins, the intermediate ions have triangular configurations which cannot be represented satisfactorily by classical bond structures. Attention is also drawn to a synthesis of violanthrone which involves an entirely novel mode of reaction, in that a base abstracts a hydrogen atom from an aromatic ring to give an anion, which may then attack a second molecule of substrate to give a dimeric product. This reaction is particularly interesting in view of the possibility that a similar abstraction of hydrogen (or an electron) may be the first step in some radical substitutions. The theoretical possibility that a *merocyanine* dye might approximate so closely to the extreme zwitterionic structure, that a decrease in the dielectric constant of the medium would actually increase the degeneracy of the dye and exert a bathochromic effect, has been realised experimentally. The colours of basic dyes have been studied by the molecular orbital method, and rules have been found relating them to chemical structure, which not only confirm earlier qualitative speculations, but also introduce the idea that electromeric substituents, or additional heteroatoms, in a cyanine dye should have dissimilar effects at positions of like or opposite polarity to the terminal basic groups. It has been shown that conjugation of two even alternant systems necessarily has a bathochromic effect; but this is not necessarily so in odd alternant systems, where hypsochromic shifts are often observed and also predicted by the molecular orbital method. An interesting empirical rule connecting colour and light absorption in a wide range of dyes is also mentioned.

An outstanding advance in stereochemistry is the rigid proof by X-ray crystallographic methods of the correctness of Fischer's guess at the absolute configuration of tartaric acid and, therefore, in principle, of all other asymmetric molecules.

In the aliphatic field, interesting developments in the chemistry of the polyacetylenes, the synthesis of the much debated vitamin A₂, and two independent total syntheses of non-benzenoid steroids are reported. There has also been much progress in the chemistry of the triterpenes and of the tropolone group of non-benzenoid aromatic compounds.

Among general methods, some novel applications of lithium aluminium hydride are given, including the direct reduction of sulphones to sulphides, which had not been accomplished previously. Also reference is made to the increasing use of *N*-halogeno-amides and -imides for the oxidation of secondary alcohols to ketones, and an interesting series of papers is noted which describes the oxidation of unsaturated compounds with nitrous

oxide at high temperatures (200–350°C.) and pressures (up to 2000 atm.).

A theoretical consideration of the coiling possibilities of peptide chains has led to the postulation of completely new configurations of the peptide backbone in protein molecules, which agree well with the experimental findings and seem likely to throw a completely new light on protein chemistry; on the experimental side, the detailed arrangement of more than two-thirds of the amino acid residues in insulin has been established.

The fourth chapter is on *Biochemistry*.

In *Analytical Chemistry* (Chapter V), a classification into sections of all the analytical publications appearing over the past year shows a high preponderance dealing with (1) colorimetric and absorptometric techniques, and (2) chromatographic and similar separations. It is pointed out that the absorptometer is now firmly established as a laboratory tool, and also that there is much scope for a proper comparison and evaluation of existing methods. It is deduced that ion-exchange, partition, and extraction methods, perhaps in novel forms, hold high promise for the solution of an extensive variety of problems in the future. It is stated that a clear case exists for increased use of statistical investigation as one of the major concerns of the analytical chemist at the present time. Analytical progress is reviewed under the headings—reagents, inorganic qualitative analysis, inorganic gravimetric analysis, inorganic volumetric analysis, classical organic analysis, and instrumental methods (including electrodeposition, coulometry, polarography, amperometric, potentiometric, and conductimetric titrations, colorimetry and absorptometry, nephelometry, fluorimetry, emission spectroscopy, visible, ultraviolet, and infrared absorption, mass-spectrum analysis, and chemical microscopy), physical separation methods (including chromatography, ion exchange, extraction processes, partition chromatography, and electrophoresis), and miscellaneous (including radiochemical, gas, and sedimentation analysis; determination of moisture; and education). There are 1319 references.

In the sixth chapter, on *Crystallography*, discussion is concentrated on protein structures only, and deals with amino acids and small peptides, types of protein fibre, X-ray patterns and infrared absorption spectra of synthetic polypeptides, structure of the α -chain, evidence relating to the α -helix, globular proteins, β -structures, collagen, muscle, nucleoproteins, and nucleic acids.

It is obvious that a volume which contains 3925 references (many of them composite) will be indispensable to chemists of all branches of the subject, but the manner of compilation deserves the highest praise and will greatly ease the quest of the reader.

H. H. HODGSON

Synthetic Methods of Organic Chemistry

An Annual Survey Volume 6

By W. Theilheimer. 1952. Pp. xi + 401. Basle and New York: S. Karger. London: Interscience Publishers Ltd. Price, Swiss francs 54.10.

This annual survey continues the collection of organic synthetic methods, and consists of abstracts

from papers published mostly in 1949 and 1950, with supplementary references from 1951. The volume is intended to be the first one of a second series, but new references are included to material in the preceding volumes, while the index also contains a few additional or revised entries to former volumes. The abstracts are limited to the information required for an appraisal of the applicability of a desired synthesis, which includes the number and nature of the reaction steps, the yield, and the important literature in question. The volumes are intended for immediate reference in the laboratory, since they provide a quick survey of the situation in hand, and obviate the necessity of first searching the entire literature, for which purpose syntheses are recorded in a subject index by starting materials and end products, along with a systematic arrangement for the methods. This has made possible a subclassification within the special reaction symbols by reagents, thereby providing a further methodical criterion. As begun in Vol. 5, a table indicating the sequence of the reagents has been included, which will help the reader to locate reactions in the body of the text, particularly within large chapters. This table also contains such frequently used reagents as NaOH and HCl, which are not included in the subject index. Another innovation is the indexing of complex compounds with cross-references under the related simpler compounds. General terms, such as synthesis, replacement, heterocyclic compounds, are also brought to the special attention of the reader. In order to carry out a particular synthesis, however, it is still necessary to have recourse to the original papers or, at least, to an abstracting journal. To avoid repetition, selections have been made on the basis of most detailed description and best yields, whenever the same method is used in similar cases. Continuations of papers already included have not been abstracted, unless they contained essentially

new information, but are quoted at the place corresponding to the abstracted papers. Papers have not been included when their content did not fit the subject of *Synthetic Methods*, and when they could be found easily in the indexes of the abstracting journals. Less accessible or readable papers, such as the Russian ones, have generally only been abstracted when the method in question is described nowhere else. An interesting utilitarian feature is the inclusion of failure as well as of success when such information is available; e.g. the cleavage of 1-hydroxy-3-methyl-7-methoxyanthraquinone occurs when it is heated with a mixture of AlCl_3 and NaCl , but not when treated with HBr in glacial acetic acid. With 911 reaction sections, this volume as well as its predecessors should be available to every organic research chemist, who, by constant use, will find them indispensable.

H. H. HODGSON

New British Standard

B.S. 1795 : 1952

EXTENDERS FOR PAINT

This British Standard incorporates all the previous specifications, viz. B.S. 255, 260, 280, 281, 301, and 926. Specifications are given for asbestine, barytes (grades 1 and 2), blanc fixe, witherite, precipitated barium carbonate, whiting, china clay (grades 1 and 2), and kieselguhr. Some of the extenders specified are also used as pigments in water paints, but the standard is not intended to cover all the requirements for materials used for that purpose. Some materials occasionally used as extenders are not included in the specification, but the methods of test laid down will help when comparing the properties and characteristics of these materials with an accepted sample. This standard can be obtained from the British Standards Institution, 24 Victoria Street, London S.W.1, price 3s. 6d.

C. O. CLARK

Abstracts from British and Foreign Journals and Patents

(The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., 68, 23 (Jan. 1952); while other abbreviations and symbols, together with a list of the periodicals abstracted, will be found at the end of the annual index.)

I—PLANT; MACHINERY; BUILDINGS

Improvements in Finishing Machinery. A. Verneuil. *Industrie text.*, 68, 379-382 (Aug.); 430-434 (Sept.); 475-477 (Oct. 1951); *J. Textile Inst.*, 43, A 402 (June 1952).

Improved designs in drying equipment—Dungler drying machine (stenter type), cylinder drying machine, tensionless drying machine, drying of printed material, carbonising machine, and polymerising machine—are described with illustrations. In dyeing by the horizontal foulard, the use of compressed air rather than hydraulic pressure on the rollers of the padding machine is found to result in more even dyeing.

C. J. W. H.

PATENTS

Even Distribution of Substances added to a Viscous Liquid, especially a Spinning Solution. N.V. Onderzoekingsinstituut Research. BP 677,081

Drying Wound Packages of Rayon, etc. American Viscose Corp'n. USP 2,589,400

The packages are carried by a foraminous support which moves in a cyclical path, along part of which a high-frequency electric field is maintained while at the same time hot air is passed up through the support and above

the packages to facilitate removal of the moisture vaporized by the dielectric heating and to prevent the vaporized moisture from condensing on the apparatus through which the support moves.

C. O. C.

Maintaining Constant Tension in Cloth, etc. J. Dilworth. BP 674,331

Constant tension is maintained in cloth, etc. as it passes through baths in which it may shrink or stretch by passing it over a roller journaled in a pair of brackets keyed on to a hollow shaft balanced by an adjustable counterweight. Fluid is applied under pressure to the bore of the hollow shaft, and passes through one of two ports in a stationary bracket to actuate mechanism operating belt guides engaging a driving belt passing over cone pulleys.

C. O. C.

Treating Long Webs of Material with Air or other Gas. E. Gordon Whiteley. BP 675,329

Improved means of ensuring uniform treatment over the whole width of the web are described.

C. O. C.

Cloth Expanders. Sir James Farmer Norton & Co. BP 674,253

An improved roller-type expander which has long life and does not damage even delicate fabrics.

C. O. C.

Felt-hardening Machine. Felters Co. *USP* 2,590,142

A machine in which the rollers can be vibrated more quickly than hitherto possible, with consequent reduction in time required for hardening. C. O. C.

Tenter Clips. Svenska Flaktfabriken A.-B. *BP* 674,347

A clip readily adjustable to deal with cloths of various thicknesses. C. O. C.

Garment Presses. United States Hoffman Machinery Corp.

BP 677,210

Treating Hides and Skins with Liquid. United Shoe Machinery Corp.

USP 2,590,194

A machine specially suitable for the "wetting back" of heavy hides is described in which successive narrow portions of the hide are worked or vibrated while in contact with the liquor. This speeds up penetration of the liquor into the leather. C. O. C.

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Non-ionic Surface-active Agents. L. Marcou. *Teinter*, 17, 421-443 (July 1952).

Review under the headings—preparation and properties of ethylene oxide derivatives; application as detergents, wetting-out, softening, antistatic, and dispersing agents; application in the dyeing of wool, of cotton with vat, azoic, direct, sulphur, and basic dyes, and of acetate rayon and nylon with disperse dyes; and use in stripping of vat and azoic dyes. B. K.

Wool Wax. II—Lime Saponification. S. D. Rousseau and E. von Rudloff. *J. Appl. Chem.*, 2, 335-338 (June 1952).

The rate of saponification of wool wax with lime has been studied, and the effect of certain additives investigated. The optimum reaction mixture was considered to be—wool wax 100, lime 9-6, water 15, alkaryl-ethylene oxide condensate 0-1, complete saponification in kg. quantities being achieved in 6 hr. at 140°C. Satisfactory separation of unsaponifiables and calcium soaps was obtained by extracting with acetone at 50°C. Ethylene dichloride gave a slightly inferior separation. W. K. R.

PATENTS

Surface-active Compounds. Basf. *BP* 676,857

Products of high wetting power are obtained by halogeno-sulphonating hydrocarbon mixtures of b.p. 120-200°C. and of chain length 8-11 C, converting the sulphonyl halides into sulphonimides, and neutralising the latter. C. O. C.

Non-foaming Surface-active Agents and Detergents.

Monsanto. *BP* 675,993

The condensates of a branched-chain *tert*-alkyl-mercaptan, derived from a polymerised olefin and containing 6-18 C, and ethylene oxide are better detergents than those obtained from straight-chain mercaptans. C. O. C.

Detergent. California Research Corp. *USP* 2,589,190

Marked increase in the detergent efficiency of soaps or synthetic detergents is obtained by adding a small amount of a celluluronate of a univalent cation. C. O. C.

Detergent. N.V. De Bataafsche Petroleum M.-S.

BP 676,449

A mixture of an alkali-metal or ammonium salt of an organic sulphonic acid having a hydrophobic group of > 7 C and 35-60% of the stoichiometric equivalent of an Mg compound of < 0.04% solubility in water, e.g. MgO, is a good detergent for cotton. C. O. C.

Solvent Scouring of Wool. F. Fiala. *BP* 675,660

Nitrated kerosene is an excellent agent for the solvent scouring of raw wool. C. O. C.

Easily Scourable Mineral Oil Blends for Textiles.

Textile Oil. *BP* 674,829

Textile fibres are lubricated by applying a mineral hydrocarbon oil, or a non-aqueous mixture of such an oil compounded with one or more saponifiable fatty full-ester oils in which the mineral oil content is > 20%, into which has been blended a partial ester (containing not > 2 free OH groups per mol.) of a polyhydric aliphatic alcohol and a monocarboxylic acid. The polyhydric alcohol must be an aliphatic glycol, a polyhydroxy-substituted saturated

or unsaturated hydrocarbon, or an ether-type glycol. The ester must contain at least one OH esterified with a wool-grease acid, and any other esterified OH groups must be esterified with an organic monocarboxylic saturated or unsaturated fatty or monohydroxy fatty acid or a cyclic petroleum naphthenic acid. One example comprises 80-95% mineral oil and 20-5% of a glyceryl diester mixture of wool-grease acids (3 mol.) with a triglyceride oil, e.g. coconut, palm, or olive (1 mol.), and glycerol (2 mol.). Such compositions are easily emulsifiable and scourable. J. W. R.

Carding Lubricant for Cellulosic Fibres. Monsanto.

USP 2,590,659

A mixture of an alkaline-reacting silica squalol, a small amount of a water-soluble or water-emulsifiable oil, and an organic hygroscopic agent is a good lubricant for application to cotton and other cellulosic fibres before they are carded. C. O. C.

Alkylene Glycol Esters of Alginic Acid. Kelco Co.

BP 676,618

The water-soluble glycol alginates obtained by treating alginic acid with 1:2 or 1:3-alkylene oxides are highly effective emulsifiers for preparing oil-in-water emulsions of animal, vegetable, and mineral oils, their efficiency as such being far superior to that of the water-soluble salts of alginic acid with various bases. C. O. C.

Thickening Starch Suspensions. Maatschappij voor Kolnbevering Stamcarbon.

BP 675,034

Liquid suspensions of maize starch are thickened by passing through one or more hydrocyclones at 40-60°C., a hydrocyclone being a radially symmetrical chamber having a tangential feed inlet for liquid material near one end, an axial discharge aperture for clarified liquor near the inlet, and an aperture for thickened suspensions remote from the inlet. J. W. R.

Stable Starch Derivatives. National Starch Products.

BP 675,793

The products obtained by treating starch with β -lactones yield aqueous dispersions of remarkable stability. C. O. C.

Amylases of Improved Enzymatic Efficiency. Kalle & Co.

BP 677,387

Addition of soya-bean amylases to the amylases produced by *Bacillus mesentericus* speeds up their rate of saccharifying starch and at the same time renders them capable of saccharifying starch to a larger extent even when only small quantities are used. C. O. C.

Aqueous Dispersions of Partly Condensed Urea-Formaldehyde Resin. British Cellophane.

BP 675,477

Urea is condensed with formaldehyde by heating in an aqueous acid medium, there being added to the medium, before or after the condensation, a water-soluble cation-active compound containing a quaternary ammonium or phosphonium or a tertiary sulphonium group and, attached directly to the N, P, or S, a straight-chain alkyl of > 7 C or a branched-chain alkyl of > 11 C. The resulting dispersions are used as water-repellent finishes. C. O. C.

Alkali-metal Salts of Organosiloxanols and Organosilanoltrichlorides for Finishing Textiles. Corles Chemical Co.

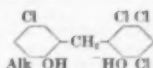
USP 2,587,636

Compounds of formula $(\text{RSi})_n\text{O}_{m+1}\text{M}_{m+1}$ (R = Ar, alkaryl, alkyl, or alkylalkyl; n > 0; M = a metal or strong organic base; m = valency of M), e.g. the product obtained by treating 1 mol. of ethylphenyltrichlorosilane with < 6 equiv. of an aqueous alkali-metal hydroxide, have wetting and detergent properties. Textiles impregnated with an aqueous solution of such a compound and then treated with acid have a silicone polymer precipitated on and in the fibres which imparts a durable, resistant, and water-repellent finish. C. O. C.

3-Alkyl-2:2'-dihydroxy-3':5':5':6'-tetrachloro-diphenylmethane Antiseptics. Dow Chemical Co.

BP 676,859

Compounds of formula—



(Alk = isopropyl or sec. butyl) are insoluble in water but somewhat soluble in many organic solvents. They have strong bactericidal and fungicidal properties, and are useful as antiseptics in detergents, plastics, rubber goods, etc. C. O. C.

Heat-stabilisers for Polyvinylidene Chloride Compositions.

Dow Chemical Co. BP 677,505
Polyvinylidene chloride compositions are stabilised to discoloration on heating by addition of 0.5–5.0% by wt. of ethylenediamine-*N,N'*-tetra-acetic acid or a salt thereof in which $>1\text{H}$ are replaced by identical alkali-metal ions and the other hydrogen ion or ions by alkaline-earth or alkali metal ions. C. O. C.

Solvents for Acrylonitrile Polymers.

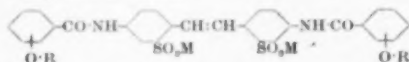
American Viscose Corp. USP 2,588,334–5
Mixtures of nitromethane and α -hydroxy- β -trichloropropionitrile, or of nitromethane (40–90% by vol.) and formic, dichloroacetic, or cyanoacetic acid (60–10), while having no swelling action on acrylonitrile polymers, dissolve them at $<80^\circ\text{C}$. C. O. C.

Whitening Nylon. American Textile Co. BP 675,156
1-Naphthylamine-4(or 5 or 7)-sulphonic acid is an optical bleaching agent having good affinity for nylon, on which it is fast to washing. C. O. C.

Optical Bleaching Agents.

General Aniline. USP 2,589,519

Compounds of formula—



(R = halogenoalkyl or halogenoalkenyl; M = H or a cation) are particularly suitable for use as optical bleaching agents in textile detergents or for incorporation in discharge printing pastes. C. O. C.

Water-dispersible Fatty Compositions for Treating Hides and Textiles.

Nopeo Chemical Co. BP 676,686
Water-dispersible mixtures of sulphated raw fats and sulphated blown fats with or without unsulphated blown fats are used as fat-liquoring agents for leather and as textile softening agents. C. O. C.

Vulcanisation Accelerators.

Monsanto. BP 676,248
Compounds of formula—



(M = H, metal, NH_4 , or Alk., *cyclo*Alk., or aralkyl-subst. NH_4 ; R^1 and R^2 = H, Alk., *cyclo*Alk., hydroxyalkyl, cyanoalkyl, aralkyl, or thiocarbamyl, but both may not be H) are capable of acting as accelerators possessing inherent weaknesses, e.g. inefficient vulcanising effect or limited solubility or poor dispersibility in rubber sticks, so giving even cures in reduced time. C. O. C.

Aluminium Salts of Organic Acids as Aids in Vulcanising with Phosphorus Sulphides.

A. Skipsey. BP 677,323
Better vulcanising with use of less phosphorus sulphide is obtained with the assistance of aluminium salts of organic acids. C. O. C.

Sighting Colour for Acetate Rayon.

Celanese Corp. of America. USP 2,590,236
A fugitive dye, e.g. C.I. 53, 670, 698, or 699, is applied in a dispersion of isobutyl alcohol (1–5% by wt.), water (90–97), and a sodium dialkyl sulphosuccinate (0.2–1.0). Such dispersion does not tend to become foul. C. O. C.

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Solvent Extraction of Phenol from Coal-tar Hydrocarbons—Use of Glycerol, Triethylene Glycol, and their Aqueous Solutions as Solvents.

A. F. C. Cumming and F. Morton. *J. Appl. Chem.*, 2, 314–323 (June 1952).
Ternary solubility data for the systems glycerol-phenol-hydrocarbons and triethylene glycol-phenol-hydrocarbons at 20°, 40°, and 60°C, and phase equilibrium data for the same systems are presented. The hydrocarbon fraction used, prepared from a high-temp. tar, had a

boiling range of 173–180°C, and an aromatic content of 67% and was free from phenols and bases. Glycerol gives the most complete separation of phenol from hydrocarbons, and a final product containing up to 99% pure phenol can be produced. Using a 1:1 solvent ratio, two theoretical stages are required to reduce the phenol content of the raffinate from 25% to $<0.25\%$. Addition of water and increase in temperature reduce the efficiency of the separation. With triethylene glycol a product of up to 92% pure phenol only can be produced. This result is similar to that obtainable with 70% methanol (Metasolvan process). Addition of water to triethylene glycol reduces the solubility of hydrocarbons in the extract, but increases the number of theoretical stages required for separation. The optimum water content is ca. 35%. Phenol is separated from the extracts by distillation; there is no evidence of azeotrope formation in either case. W. K. R.

Mononitration of o-Xylenes.

K. A. Kobe and P. W. Pritchett. *Ind. Eng. Chem.*, 44, 1398–1401 (June 1952).
A maximum yield of 90% mononitro-o-xylenes has been obtained using the following conditions—sulphuric acid : o-xylene weight ratio 2:15, initial concentration of sulphuric acid 79.4%, nitric acid : o-xylene mole ratio 1:16, temp. 25°C, nitration time 30 min. The product consisted of 58% 3-nitro- and 42% 4-nitro-o-xylene. With more dilute acid, higher temp. can be used to give equal yields. W. K. R.

Decarboxylation of Anthranilic Acid.

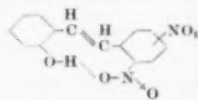
W. H. Stevens, J. M. Pepper, and M. Lounsbury. *Canadian J. Chem.*, 30, 529–540 (July 1952).
Anthranilic acid is decarboxylated by heating above its m.p. or by boiling in water. Addition of mineral acid catalyses the aqueous decomposition, but after the concentration of the catalysing acid approximates to that of the anthranilic acid the reaction decreases with further addition. Mass spectrometer study of liberated CO_2 shows that ^{13}C -carboxyl anthranilic acid decomposes at the same rate as ^{12}C -carboxyl anthranilic acid; thus there is no "isotope effect" as with some other organic acid decarboxylations. The mechanism of decarboxylation is explained as bimolecular electrophilic substitution with the attack of a proton the rate-determining step. Proton attack on the α -carbon of the zwitterion is suggested as the most probable mechanism. A. H.

Steric Effects on Mesomerism. VII—Decacylation of Acetylaminonitronaphthalenes.

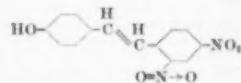
L. H. Krol, P. E. Verkade, and B. M. Wepster. *Rec. Trav. chim.*, 71, 545–564 (May 1952).
The rates of decacylation by means of sodium methoxide of a number of acetylaminonitronaphthalenes are measured, and discussed in terms of the limiting mesomeric forms of naphthalene and its derivatives. Heteronuclear acetylaminonitronaphthalenes give predicted results. This is also the case with homonuclear compounds with the exception of 2-acetylamino-1-nitronaphthalene, which shows no steric inhibition of mesomerism. The decacylation rates of derivatives with two substituents in the *peri* position cannot be satisfactorily explained. A. J.

Intramolecular Hydrogen Bonding—Ten-membered Rings.

H. Hoyer. *Kolloid-Z.*, 126, 52–53 (April 1952).
To discover whether rings of >6 atoms can be formed by hydrogen bonding, compound I (*cis* or *trans* uncertain)



2-Hydroxy-2'-nitro-1,1'-dinaphthalene (m.p. 154°C.)
(I)



4-Hydroxy-2'-nitro-1,1'-dinaphthalene (m.p. 161°C.)
(II)

was synthesised from salicylaldehyde and trinitrotoluene in the presence of piperidine. In solution in benzene I runs faster on a silica gel column than II; the OH group of I is more easily acetylated than II; in CCl_4 I has no OH frequency in the infrared at 2.74 μ , but II has. These properties, characteristic of 6-membered H-bond ring formation, show that similar 10-membered rings are possible. L. P.

Influence of Substituent Groups on Nuclear Reactivity in Formation of Substituted Diazobenzenes through Reactions of Aromatic Diazo and Cognate Compounds with Aromatic Liquids.

II—The Bromine Atom. O. Simamura, T. Inukai, and M. Kurata. *Bull. Chem. Soc. Japan*, **25**, 76-78 (Feb. 1952).

N-Nitrosoacetanilide and benzenediazonium hydroxide both react with bromobenzene giving a mixture of *o*-, *m*-, and *p*-bromodiphenyls, in the average ratio 57 : 25 : 18. In competition reactions with 2 parts of benzene and 1 part of bromobenzene, the ratio of diphenyl to bromodiphenyl is 1 : 1.54, giving reactivities of 5.7, 2.5, and 2.7 to the *ortho*, *meta*, and *para* positions of bromobenzene relative to one position in benzene as unity for homolytic reactions with the phenyl radical. A. J.

Action of Light on Diazoaminobenzene dissolved in Ethanol.

H. C. Freeman and R. J. W. Le Fevre. *J.C.S.*, 2932-2934 (July 1952).

Although Le Fevre and Vine showed that the dielectric constants of solutions of diazoaminobenzene in benzene were not seriously affected by illumination, the spectroscopic behaviour of alcoholic solutions of diazoaminobenzene, when examined before and after illumination for varying periods, and again after storage in darkness, indicates decomposition to take place rather than a photochemically reversible change. H. H. H.

Aspects of the Chemistry of Azo Colouring Matters.

A. Burawoy. *J. Oil & Col. Chem. Assoc.*, **35**, 298-313 (July 1952).

Three aspects of the chemistry of azo dyes are discussed—(1) Mechanism of diazotisation, where it is shown that at very low concn. of mineral acid (0.002 N), the factor determining the rate of reaction is formation, from 2 mol. of HNO_3 and Na_2O_2 , which is quickly removed by the free amine present. The reaction is bimolecular and independent of the concn. and nature of the amine. At higher acid concn. the reaction between free amine and Na_2O_2 becomes rate-determining, and the kinetics changes to one of third order. With HCl or HBr (in contrast to oxyacids) a second dominating mechanism follows a third-order kinetics and prevails at acid concn. above approx. 0.01 N ; it corresponds to a reaction between the free amine and NOCl or NOBr . The effects of pH and nature of acid and amine on diazotisation rate are discussed. (2) The fundamental property of naphthalene *o*-hydroxyazo compounds, i.e. low acid strength, is ascribed to the structural characteristics of these substances, which are shown to exist generally as phenylhydrazones of 2-naphthoquinone. (3) Methods of preparation and some properties of hitherto unknown *o*-mercaptoazo compounds are discussed. A. H.

Certified Azo Colours in Cosmetics.

S. Zuckermann. *Drug & Cosmetic Ind.*, **70**, 192-193, 277-281 (1952); *Chem. Abs.*, **46**, 6321 (10 July 1952).

A review of the chemistry of azo dyes, which form over 90% of the total volume of certified dyes. C. O. C.

Some Metal Derivatives of Azo and Azomethin Dyes.

C. F. Callis, N. C. Nielson, and J. C. Bailar. *J. Amer. Chem. Soc.*, **74**, 3461-3464 (20 July 1952).

Study of the Co and Ni lakes of phenylazo- β -naphthol, 5-chloro-2-hydroxyphenylazo- β -naphthylamine, *o*-hydroxybenzylidene-2-hydroxy-5-chloroniline, and 4-*o*-carboxyphenylazo-1-phenyl-3-methylpyrazol-5-one shows that the tendency for the metal to utilise completely both the primary and secondary valences is very strong and determines the composition of the complex, even to the extent of determining the oxidation state of the metal. Magnetic measurements indicate that the Ni lakes may be either planar or tetrahedral, depending upon the nature of the dye. The azo and azomethin groups each occupy one co-ordination position. C. O. C.

Reduction of some Chlorinated Azobenzenes with Titanous Sulphate.

F. Fawcett, J. L. Myers, and K. F. Kuretsch. *Canadian J. Chem.*, **30**, 498-499 (June 1952).

The method of Knecht and Hibbert has been utilised in an attempt to analyse some chlorinated azobenzenes. It is found that only two instead of the expected four equivalents of titanous sulphate are required when 2,2'-dichloroazobenzene and 2,3-dichloroazobenzene are reduced. Experiments with azobenzene also require only two equiv. of $\text{Ti}_2(\text{SO}_4)_3$. On isolation, the reduction products are found to be benzidine—rather than aniline-type compounds. The explanation of this is a benzidine rearrangement of the intermediate hydrazo compound favoured by the strongly acid reduction medium. "Blocking" the *para* positions of azobenzene to prevent rearrangement produces normal reduction and consumes four equivalents of $\text{Ti}_2(\text{SO}_4)_3$. A. H.

Hydrolysis and Oxidation Reactions of 1-Phenylazo-3,4-dihydroxynaphthalene.

O. M. Shenyakina, B. M. Bogdanovsky, and M. M. Shenyakin. *J. Gen. Chem. U.S.S.R.*, **22**, 675-679 (April 1952).

1-Phenylazo-3,4-dihydroxynaphthalene is tautomeric with the monophenylhydrazones of 3-hydroxy-1,4-naphthoquinone, and it was surmised that it would give the hydrolysis reactions, leading to the opening of the quinonoid ring, that are characteristic of hydroxy-*p*-benzoquinone and its polynuclear analogues. It is found that, on boiling the azo cpd. in alkaline soln. in absence of air, it is indeed the quinonoid tautomer that reacts, but hydrolysis occurs first at the hydrazine linkage, giving the hydroxyquinone, which is then split hydrolytically in the manner already studied, giving e.g. *o*-acetylphenylglyoxylic acid. In presence of air, however, reaction is with the azo tautomer, which is rapidly oxidised to 1-phenylazo-3,4-naphthoquinone. A. E. S.

Cinnolines and other Heterocyclic Types in relation to the Chemotherapy of Trypanosomiasis.

III—Synthesis of 4:4'-Diamino-6:6'-azocinnoline Metho-salts. P. E. Maery and J. C. E. Simpson. *J.C.S.*, 2602-2605 (July 1952).

IV—Synthesis of Azocinnoline Derivatives. J. McIntyre and J. C. E. Simpson. *Ibid.*, 2606-2615 (July 1952). V—Quaternary Salts of 4:4'-Bismethylamino-6:6'-azocinnoline. J. McIntyre and J. C. E. Simpson. *Ibid.*, 2615-2616 (July 1952).

Effects of Viscosity and Temperature on Fluorescence of Dye Solutions.

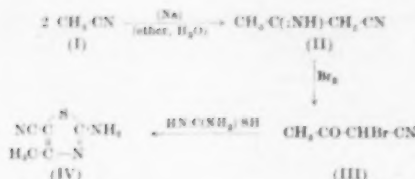
N. R. Tawde and N. Ramasathan. *Proc. Phys. Soc. B*, **65**, Part 1, 33-40 (1952); *J. Textile Inst.*, **43**, A 405 (June 1952).

The effects of viscosity and temperature of the medium on the polarisation of the fluorescent light emitted by some dye solutions when excited by linearly polarised monochromatic light are investigated by visual and photoelectric methods. The variations in the polarisation caused by temperature are found to depend on the concentration and molecular weight of the dye. C. J. W. H.

Synthesis of 2-Amino-5-cyano-4-methylthiazole from acetonitrile.

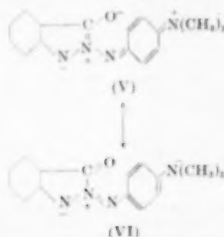
K. Murata. *Bull. Chem. Soc. Japan*, **25**, 16-18 (Feb. 1952).

β -Iminobutyronitrile (II) is prepared from acetonitrile (I) by the action of sodium in absolute ether soln. at 40°C. The ppt. thus obtained is shaken with ether and water, and the ether soln. concentrated and cooled with ice for 2-3 days, when crystals of II are obtained. Repeated recrystallisation from benzene gives a stable form, m.p. 47-48°C. A labile form, m.p. 74-78°C., is also obtained. II is treated with bromine at 0°C. in ether soln., the excess Br_2 removed with NaHSO_3 , and the soln. dried with Na_2SO_4 and evaporated. The resulting oil is separated from NH_4Br and distilled *in vacuo*, giving *o*-bromo- β -keto-butyronitrile (III), b.p. 70-72°C./4 mm. Hg. The theoretical amount of thiourea is added to an aq. soln. of III at 80°C. and left overnight, water is added to dissolve any ppt., unreacted material is extracted with ether, alkali added to the aq. soln. to pH 6-8, and the resulting ppt. of 2-amino-5-cyano-4-methylthiazole (IV) filtered and recrystallised from water. M.p. 104-105°C.; soluble in benzene, alcohol, and hot water.



New Class of Magenta Couplers. J. Jenner. *Ind. chim. belg.*, **16**, 472-474 (1951); *Chem. Abs.*, **46**, 6387 (10 July 1952).

In attempts to prepare 3-aminopyrazolocoumarone (I) from $\text{C}_6\text{H}_5 \cdot \text{O} \cdot \text{CNH}_2$; $\text{CH}_3\text{COOC}_6\text{H}_5$ and $\text{HOOC} \cdot \text{C}_6\text{H}_4 \cdot \text{NH} \cdot \text{NH}_2$ (II), and the 3-hydroxy analogue of I from malonic ester and II, indazole (III) is obtained as the only product. Similarly to the coupling of pyrazolones in the *ortho* position to the end group, III couples with $p\text{-(CH}_3)_2\text{N} \cdot \text{C}_6\text{H}_4 \cdot \text{NO}$ (IV) to give a blue dye of the mesomeric structures V and VI. III gives with conc. H_2SO_4 a yellow solution turning magenta on dilution with



water. The analogous dye obtained by coupling 4,6-dimethyl-1H-pyrazolo[4,3-c]pyridine-3(2H)-one with IV gives a blue with 1 mol. of acid. C. O. C.

Dyeing Concept of Dyes. L. G. S. Brooker. *Symposium on Molecular Structure and Spectroscopy* (Ohio State University, 9-13 June 1952); *Anal. Chem.*, **24**, 1224 (July 1952).

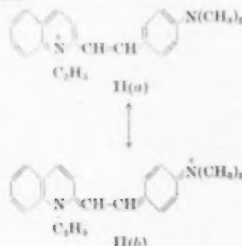
Though long conjugated chains of carbon atoms are necessary for the appearance of deep colour in dye molecules, they are not always sufficient in themselves. For deep colour it is necessary for the linkages to be reversible in the sense—



i.e. the two resonance forms should be of as nearly the same energy as possible. In symmetrical cyanines, for example



I(a) and I(b) are identical, and the conjugated linkages are completely delocalised. In the following, which is unsymmetrical



II(a) is of lower energy than II(b) (and thus predominates), and the absorption lies at appreciably shorter wavelengths (by an amount called the deviation in λ_{max}) than if they were of equal energy.

Deviations, worked out for many dyes, provide a means of classifying the dyes; the smaller the deviation the greater the delocalisation of the linkages, and the deeper the colour of the dye, other things being equal. In some cases at least, the intensity of absorption appears to reach a max. value in energetically symmetrical dyes.

J. W. D.

Determination of Crystal Structure [of Purpuragallin] by Optical Diffraction Methods. I—A. W. Hanson, C. A. Taylor, and H. Lipson; II—C. A. Taylor; III—J. D. Dunitz. *Nature*, **169**, 1086-1088 (28 June 1952).

I—Structures of *p*-diisocyanobenzene, diphenylphthalene, thiourea dioxide, and di-*p*-anisyl nitric oxide are determined fairly simply by the Fourier-transform method, which is considered to be a more powerful tool than optical Fourier synthesis.

II—An account of optical diffraction methods applied to purpuragallin.

III—X-Ray Fourier synthesis of purpuragallin.

J. W. H.

Aromatic Keto-enols. II—Some New 2:3-Dihydro-1:4-naphthaquinones and -anthraquinones. D. B. Bruce and R. H. Thomson. *J.C.S.*, 2759-2766 (July 1952).

Several new 1:2:3:4-tetrahydro-1:4-diketonaphthalenes are described which are obtained either by fusion of the dienol or reduction of the corresponding 1:4-naphthaquinone with acid stannous chloride. The elimination of β -substituents, well known in the anthraquinone series, is observed with juglone (5-hydroxy-1:4-naphthaquinone) and naphthazarin derivatives; thus, in the former series elimination occurs when a β -substituent is OH , $\text{N}(\text{CH}_3)_2$, $\text{NH} \cdot \text{C}_6\text{H}_5$, or $\text{S} \cdot \text{C}_6\text{H}_5$, and in the latter series when a 2-Cl group is present, though a 2- CH_3 , 2- OH , or 2- $\text{NH} \cdot \text{C}_6\text{H}_5$ is retained. 1:2:3:4-Tetrahydro-1:4-diketanthracene is obtained in 50% yield by fusion of 1:4-dihydroxyanthracene, but no change is observed after fusion of 1:4-naphthylendiamine. The structure of leuco-1:4:5:8-tetrahydroxy-anthraquinone is discussed. H. H. H.

Action of Benzoyl Peroxide on Polycyclic Aromatic Hydrocarbons. I. M. Rott and W. A. Waters. *J.C.S.*, 2695-2705 (July 1952).

Anthracene, 1:2-benzanthracene, and 3:4-benzopyrene are attacked in their exposed *meso*-positions, and benzoate groups ($\text{C}_6\text{H}_5\text{COO}$) are substituted for hydrogen, by benzoyl peroxide at 80°C. in chlorobenzene under nitrogen; phenanthrene and chrysene are unattacked, and 1:2:5:6-dibenzanthracene reacts only slowly in air. Under similar conditions 9-methylanthracene is very reactive. These experimental results are discussed in relation both to concepts of homolytic reactivity and to previously known chemical and biochemical oxidations of these hydrocarbons. H. H. H.

Formation of Flavanthrones from Derivatives of 2-Aminoanthraquinone. W. Bradley and H. E. Nursten. *J.C.S.*, 2944 (July 1952).

When 2-amino-1-bromo-3-*tert*-butylanthraquinone is heated with sodium and copper acetates for only 2 hr., 3:3'-di-*tert*-butylindanthrone, 3:3'-di-*tert*-butylindanthranthone, and 2-amino-3-*tert*-butylanthraquinone are formed; neither 2:2'-diamino-3:3'-di-*tert*-butyl-1:1'-dianthraquinonyl nor 3:3'-di-*tert*-butylflavanthronone is present in the product. When, however, an equimolecular mixture of 2-amino-3-*tert*-butylanthraquinone and its 1-bromo derivative is heated under the same conditions, the formation of 3:3'-di-*tert*-butylflavanthronone again takes place. The result suggests that the formation of the flavanthrone involves two reactions, viz. initial debromination, and condensation of the debrominated product with unchanged bromo derivative. H. H. H.

Electrical Conductivity of Condensed Polynuclear Aromatic Compounds. H. Inokuchi. *Bull. Chem. Soc. Japan*, **24**, 222-226 (Nov. 1951).

The resistivity of the following eleven compounds in the compressed (80 kg./sq. cm.) powdered state is measured by the potential drop method at temp. up to 300°C.—isoviolanthrone, isoviolanthrone, violanthrone, violanthrone,

ovalene, pyranthrone, pyranthrene, mesomaphthodianthrone, mesomaphthodiantrene, anthanthrone, anthanthrene. Resistivity ρ can be expressed by the relation $\rho = \rho_0 \exp (AE/2kT)$, and the compounds are considered to be semi-conductors. Both ρ and AE decrease with increasing number of π electrons or conjugated double bonds in the molecule, from $\rho_{11} = 1.5 \times 10^{10}$ ohm-cm., $AE = 1.67$ e.v. for anthanthrene to 5.7×10^8 ohm-cm. and 0.75 e.v. for α -violanthrone. A. J.

Electrical Conductivity of Condensed Polynuclear Aza-aromatic Compounds. H. Inokuchi. *Bull. Chem. Soc. Japan*, **25**, 28-32 (Feb. 1952).

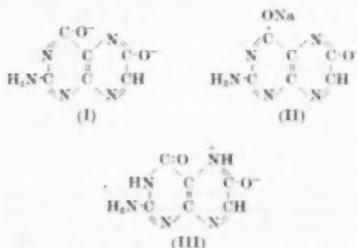
The resistivity of the following seven nitrogen-containing polynuclear compounds in the compressed, powdered state has been measured at temp. up to 300°C.: cyananthrone, indanthrone black, flavanthrone, indanthrone, indanthrazine, 5,6(N)-pyridino-1,9-benzanthrone, and 1,9,4,10-anthradipyrimidine. In all cases resistivity may be expressed by $\rho = \rho_0 \exp (AE/2kT)$, and the compounds are regarded as semi-conductors. The resistivities are much smaller than those of the related aromatic compounds. A. J.

Melanin and its Precursors. V—Synthesis of 5- and 7-Hydroxyindole from Dihydroxyphenyl-alanines. R. I. T. Cromarty and J. Harley-Mason. *J.C.S.*, 2525-2527 (July 1952).

Oxidation of 2,5-dihydroxyphenylalanine, or of 2-(2,5-dihydroxyphenyl)ethylamine, at room temp. with potassium ferriyanoide in the presence of NaHCO_3 gives 5-hydroxyindole in 85% yield. Similar oxidation of 2,3-dihydroxyphenylalanine gives 7-hydroxyindole in 20% yield, whereas the methyl ester gives methyl 7-hydroxyindole-2-carboxylate. H. H. H.

Xanthopterin. IV—Fluorescence of Xanthopterin Adsorbates. K. Weber and J. Hojman. *Bull. Soc. chim. Belgrade*, **13**, 27-38 (1950); *Chem. Abs.*, **46**, 3976 (10 July 1952).

The fluorescence of xanthopterin adsorbed on various substrates, including filter paper and proteins, has been studied. Long exposure of dry adsorbates to ultraviolet radiation ($\lambda = 300-400 \text{ m}\mu$) in presence of oxygen causes loss of fluorescence owing to photochemical oxidation of xanthopterin; the fluorescence is stable if the adsorbates are kept *in vacuo*. At pH 11-2 solutions of xanthopterin have strong green, at pH 7 blue, at pH 3 yellow, and in conc. H_2SO_4 weak red fluorescence. Adsorbed on filter paper, Al_2O_3 , SiO_2 , and proteins from alkaline solution, xanthopterin has blue fluorescence, but on Al_2O_3 and SiO_2 from neutral or acid solution green and yellow fluorescence respectively. The green fluorescence is caused by the bivalent (I), the blue by the univalent (II) anion of the enolic form of xanthopterin, and the yellow fluorescence by the mesomeric form (III). C. O. C.



Arenicochrome, a New Pigment from *Arenicola marina* L. P. van Duijn. *Rec. Trav. chim.*, **71**, 585-594 (May 1952).

Arenicochrome occurs as green granules in the epithelial cells of the worm *Arenicola marina* L. It occurs only in black worms from mud rich in FeS_2 and not in "red" worms. The pigment is extracted by ammoniacal alcohol containing some ammonium oxalate and potassium cyanide, the extract chromatographed on Al_2O_3 , and the blue-green pigment eluted with aqueous alcohol. Addition of alkaline KCl gives separation of violet crystals. Analysis indicates a formula of $(\text{C}_{21}\text{H}_{12}\text{S}_2\text{K}_2\text{O}_{14})_n$ ($n = 1$ or 2). The

pigment behaves as an indicator, changing from blue in alkali to orange at pH 5.7. Reduction with $\text{Na}_2\text{S}_2\text{O}_4$ at pH 8.8 gives a yellow compound which is reoxidised by atmospheric oxygen to a blue compound of different absorption spectrum from the original. Aq. soln. are rapidly decolorized by sunlight, bromine water, dil. KMnO_4 , and H_2O_2 , and at pH 7-8 addition of Mg, Fe, and Cu salts gives a green colour. A. J.

Arenicochrome—Degradation to Arenicochromine. P. van Duijn. *Rec. Trav. chim.*, **71**, 595-600 (May 1952).

Acidification of arenicochrome in 60% ethanol with 2.8 HCl gives a purple crystalline solid containing no sulphur. This substance is termed arenicochromine, and analysis gives $\text{C}_{21}\text{H}_{12}\text{O}_{14}$ or $\text{C}_{21}\text{H}_{12}\text{O}_{13}$, decomposing at ca. 300°C. Arenicochromine behaves as an indicator, changing from red-brown to green at pH 5-6, and green to purple at pH 8-9. Arenicochromine may be a sulphate ester of a carotenoid, hydroxyquinonoid, flavonoid, or anthocyan pigment. A. J.

Flavonoids and Related Compounds. VII—Formation of 4:6:3:4-Tetrahydroxy-2-benzylcoumaran-3-one by Sodium Hydrosulphite Reduction of Quercetin. T. A. Geisman and H. Lischner. *J. Amer. Chem. Soc.*, **74**, 3001-3004 (20 June 1952).

Dyeing Constituents in "Yama-Kariyasu". M. Miyoshi. *J. Chem. Soc. Japan* (Pure Chem. Sectn.), **72**, 705-707 (1951); *Chem. Abs.*, **46**, 5848 (25 June 1952).

A flavone glycoside believed to be luteolin glucoside is one of the dyeing constituents present in the aqueous extract of the leaves of "Yama-Kariyasu", a gramineae, which have long been used in Japan for dyeing yellows. C. O. C.

Study of the Effect of a Few Groups on the Substantivity of Curcumin. D. J. Rebeiro, A. N. Kothare, and V. S. Nadkarny. *J. Univ. Bombay, Sectn. A*, **19**, Pt. 3 (Science, No. 28), 38-33 (1950); *Chem. Abs.*, **46**, 6387 (10 July 1952).

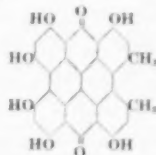
Curcumin has been acetylated, brominated, and coupled with diazotised amines to try to increase its colour value and improve its fastness. The dyeing properties of the derivatives are detailed. C. O. C.

Synthesis of Hypericin. H. Brockmann and F. Kluge. *Naturwissenschaften*, **38**, 141 (1951); *Chem. Abs.*, **46**, 5571 (25 June 1952).

1-Bromoethanol trimethyl ether was converted to the 1:1'-dianthraquinonyl derivative by treatment with CHCl_3 , and demethylated with pyridine hydrochloride to 2:2'-dimethyl-4:4':5:5':7:7'-hexahydroxy-1:1'-dianthraquinone. C. O. C.

Constitution of Hypericin. H. Brockmann, E. H. von Falkenhausen, R. Neef, A. Dorlar, and G. Budde. *Chem. Ber.*, **84**, 865-887 (1951); *Chem. Abs.*, **46**, 5567 (25 June 1952).

Evidence is given establishing the constitution of hypericin to be—



C. O. C.

Actinomycin. II—Studies on the Chromophoric Grouping. A. W. Johnson, A. R. Todd, and L. C. Vining. *J.C.S.*, 2672-2679 (July 1952).

The molecular weight of the red quinonoid antibiotic actinomycin B has been revised on the basis of quantitative hydrogenation to 1240 ± 20, corresponding to an approximate formula $\text{C}_{21}\text{H}_{14}\text{O}_{10}$. Hydrolysis with hot aq. Ba(OH)_2 gives an insoluble purple Ba salt, which on acidification yields the red peptide-free quinone, actinomycinol B, $\text{C}_{21}\text{H}_{12}\text{O}_{10}$, in which the original chromophoric system of actinomycin has been modified. The formulation of this compound as a substituted anthraquinone is shown to be untenable, and other structures containing a heterocyclic nitrogen atom are discussed. H. H. H.

Grinding and Dispersion of Titanium Pigments in Ball Mills. A. Bowman. *J. Oil & Col. Chem. Assoc.*, 35, 314-328 (July 1952).

The manufacture of titanium oxide pigments is briefly described, showing how precipitation and calcination conditions affect the properties of the product. The theory of incorporation of the pigment into paint media is discussed in terms of wetting, mechanical attrition, and true dispersion. The action of the ball mill is described, and illustrations are given of the effect of ball size, time, viscosity, and rheological properties of the mill base on the efficiency of grinding. A. H.

Dichroism of Planar Complexes. III—Colour and Structure of the Crystals of Tetracyanoplatinate (II). S. Yamada. *Bull. Chem. Soc. Japan*, 24, 289 (Dec. 1951).

Correction to previous paper, *ibid.*, 24, 127 (July 1951); *J.S.D.C.*, 68, 98 (March 1952).

Properties and Uses of Colloidal Carbon. C. W. Sweitzer, L. J. Venuto, and R. K. Estelow. *Paint, Oil, Chem. Rev.*, 115, (8), 22-24, 26, 28, 30, 32, 35, 58-60, 62, 64 (1952); *Chem. Abs.*, 46, 6361 (10 July 1952).

Colloidal carbon includes all carbon pigments of approx. 13-150 m μ . Raw materials, manufacturing processes, and conditions of manufacture affect particle size, shape, porosity, pH, chemical activity, etc. Type of process has greatest influence. Electron micrographs show that all forms have a reticulate chain structure. Tremendous surface area, as much as 12 acres/lb., accounts largely for their effectiveness in reinforcing rubber. The pH, porosity, and adsorptivity may have beneficial or harmful effects on vulcanisation and end properties. Formation of carbon sols involves wetting by the vehicle, deflocculation by mechanical means, and stabilisation by peptising agents. Wetting is less difficult in organic vehicles. Acid types wet more easily in water. Dispersing agents, peptisers, and protective colloids are usually added before deflocculation. Adsorptivity is affected by structure, porosity, oxygen-containing volatile matter, unsaturation, and alkali salts from cooling water. Properties and uses are discussed. C. O. C.

Metallic Powders and Pastes as Pigments. V. J. Hall. *Paint, Oil & Col. J.*, 122, 137-141 (18 July 1952).

A brief history of the development of metallic pigments and the two chief methods of production, viz. stamping of sheet metal and ball-milling of atomised powders, is followed by a review of the properties of the pigments and of their testing and a summary of the salient features of media formulation for use with such pigments as aluminium and the bronzes. C. O. C.

Inorganic Pigments. I—Three Types of Cobalt Orthophosphate and Relations between them.

II—Cobalt Green—Reaction between Cobalt Compounds and Zinc Oxide. T. Ando, S. Minami, and T. Tsubota. *Bull. Osaka Ind. Research Inst.*, 1, 63-69 (1950); 2, 7-11 (1951); *Chem. Abs.*, 46, 6401 (10 July 1952).

I—Pink $\text{Co}_3(\text{PO}_4)_2 \cdot 8\text{H}_2\text{O}$ was precipitated by treating a cold solution of Co salts with Na_2HPO_4 ; similar treatment of a hot solution yielded deep violet $\text{Co}_2(\text{PO}_4)_3 \cdot 4\text{H}_2\text{O}$, which after 4-5 days changed to the former pink salt in the solution. The particle size of the latter is the smaller. Heat balance studies showed that the pink salt lost $6\text{H}_2\text{O}$ at 140°C . and the deep violet salt $1.5\text{H}_2\text{O}$ at 160°C . Both salts changed to violet $\text{Co}_2(\text{PO}_4)_3$ at 600°C . and to a reddish compound at 900°C ., which was shown by the X-ray powder method to be identical with the violet anhydrous compound.

II—A study of the conditions for preparing cobalt or Ruman's green. A mixture of CoCO_3 and ZnO (1:10 mol.) was heated to $800-1100^\circ\text{C}$. for 5 hr. The products were analysed for unchanged Co_2O_3 and the reaction rates calculated. CoSO_4 , $\text{Co}(\text{NO}_3)_2$, and $\text{Co}(\text{OH})_2$ can also be used. CoSO_4 reacted most easily at about 850°C . The activation energy of the diffusion process in the formation reaction of $\text{CoO} \cdot 10\text{ZnO}$ was calculated to be 34 kcal. at $1000-1100^\circ\text{C}$. for CoCO_3 . 20 references. C. O. C.

Improvements in Colour Pigments. J. K. Donohue. *Paint, Oil, Chem. Rev.*, 115, (8), 20-21 (1952); *Chem. Abs.*, 46, 6401 (10 July 1952).

Most advances in pigments in recent times have been

improvements in existing materials rather than new types. Improvements in molybdate orange, phthalocyanine blues, chrome yellows, iron blues, organic reds, and organic molybdenum pigments are described. C. O. C.

Modern Pigments for Automotive Finishes. D. B. Kilian. *Paint, Oil, Chem. Rev.*, 115, (8), 14-16, 18 (1952); *Chem. Abs.*, 46, 6401 (10 July 1952).

Development of improved pigments has contributed to the trend toward brighter and more varied colours in car body finishes. Rutile has made possible a wide variety of tinted finishes with good gloss retention, chalk resistance, and durability. Hydrated iron oxides have proved to be good bases for transparent, metallised finishes where the tendency to fade is accentuated. Phthalocyanine pigments, blues and greens, are the outstanding new pigments from the standpoint of colour fastness, tinting strength, and chemical and heat stability. Types of inorganic and organic pigments are tabulated and evaluated. C. O. C.

PATENTS

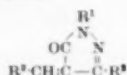
1:4-Naphthaquinone Derivatives. Research Corp.

USP 2,564,967

Improved yields are obtained by the method of preparing 2-substituted 1:4-naphthaquinones described in *USP Appl.*, 140,330 (1950) if water is omitted from the acetic acid-chromium trioxide oxidation mixture. Thus CrO_3 is added slowly to a suspension of 2-octadecyl-1-naphthol in glacial acetic acid at 50°C . The resulting 2-octadecyl-1:4-naphthaquinone is separated by adding water. R. K. F.

Colour Couplers. Ilford. BP 675,797

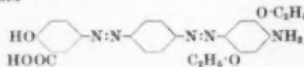
Compounds of formula—



(R^1 = an aromatic residue containing at least one sulphonic acid radical; R^2 = Alk of 10-18 C; R^3 = a phenyl or naphthyl radical containing *para* to its linkage a dialkylamino group) yield magentas with aromatic primary amino developers. C. O. C.

Metallisable Trisazo Direct Cotton Navy Blue and Black Dyes. Fby. BP 676,114

Trisazo dyes $\text{A-N-N-B-N-N-C-N-N-D}$ (A = an *o*-hydroxycarboxylic acid radical of the benzene series; B = sulbut, or unsulbut, benzene radical; C = the radical of a quinol dialkyl ether; D = the radical of an amino-naphtholmonosulphonic acid or an N-alkyl or N-aryl derivative thereof; A, B, and C are free from sulphonyl groups) are navy blue or black direct cotton dyes, which may be aftertreated on the fibre with Cr or Cu salts. Thus the monosazo compound *p*-nitroaniline-salicylic acid is reduced with Na_2S to the aminomonosazo compound, which is diazotised and coupled with aminoquinol diethyl ether under weakly acid conditions to give the aminodisazo compound—



This is diazotised and coupled in presence of soda ash with 2-amino-8-naphthol-6-sulphonic acid to give a black direct cotton dye, rendered fast to washing by aftertreatment on the fibre with Cr or Cu salts. E. 8.

Hydroxyalkylaminoanthraquinones containing a Tertiary Carbinol Group—Dyes for Wool and Cellulose Esters. Publike Industries.

USP 2,563,144

Anthraquinone derivatives containing replaceable groups in one or more α -positions are condensed with an amine of formula $\text{H}_2\text{N-CHR}^1\text{-CR}^2\text{-OH}$ (R^1 = Alk of 1-4 C; R^2 and R^3 = Alk, Ar, or are combined in a cycloaliphatic ring) to give dyes for cellulose acetate or, where the starting anthraquinone compounds contain $-\text{SO}_2\text{H}$, dyes for wool. Tinctorial value and light, washing, and sublimation fastnesses are all better than with the corresponding dyes containing hydroxyalkylamino groups which are primary or secondary carbinols. Thus 1-chloroanthraquinone is refluxed for 8 hr. in pyridine with 3-amino-2-methyl-2-butanol to produce a red dye for cellulose acetate. R. K. F.

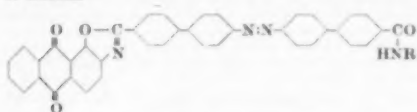
Colouring Matters containing Hydroxybenzyl Group(s). ICI. BP 675,863

Coloured compounds of the polycyclic ketone, azo, perylene, and diazine series are treated first with dichlorodimethyl ether, and the resulting chloromethyl derivatives are condensed with a phenol. The hydroxybenzyl derivatives so formed are useful for colouring textiles, paper, and lacquers, and are converted into other colouring matters by coupling with diazotised amines, or sulphonating, both reactions occurring in the hydroxybenzyl nucleus. Thus bischloromethylacridanthrone, prepared by treating acridanthrone with dichlorodimethyl ether in presence of H_2SO_4 , is heated in phenol containing $ZnCl_2$ at 120–125°C. for 2 hr.

R. K. F.

Anthraquinone Oxazole Vat Dyes containing an Azo Group. DuP. BP 675,149

Bright yellow vat dyes of good tinctorial strength and of formula—

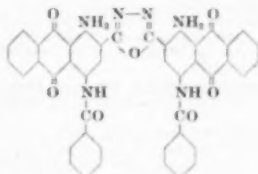


(R = vatable anthraquinonyl radical) are made by condensing 4,4'-azodiphenyl-4''-dicarboxylic chloride first with a 2-amino-1-halogeno- or 1,3-dihalogenoanthraquinone, then with an aminoanthraquinone, and finally cyclising to the oxazole; or the cyclisation may precede the condensation with the aminoanthraquinone. Thus sodium 4,4'-azodiphenyl-4''-dicarboxylate is heated at 95°C. for 12 hr. with $SOCl_2$ in nitrobenzene containing pyridine. After aerating to remove excess $SOCl_2$, 1-chloro-2-aminoanthraquinone is added, and heating continued at 135–138°C. for 6 hr. Water is then added to hydrolyse the remaining $COCl$ group, and the whole heated at 210°C. for 4 hr. after adding Na_2CO_3 , K acetate, $CuCl_2$, and Cu acetate, to cyclise to the oxazole. After separating, the resulting compound is suspended in nitrobenzene and treated with $SOCl_2$ and pyridine at 138–140°C. for 12 hr.; the acid chloride is separated and heated at 210°C. for 2 hr. with 1-aminoanthraquinone in nitrobenzene containing pyridine.

R. K. F.

Dianthraquinonyl-1:3:4-oxadiazoles—Vat Dyes. ICI. BP 676,699

Blue vat dyes faster to bleaching and of better dyeing properties than members of the indanthrone series—e.g. they can be dyed at 95°C.—are made by diacylating 2:5-di-(1:4-diamino-2-anthraquinonyl)-1:3:4-oxadiazoles. Thus the dyes—

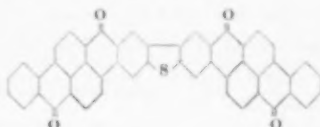


is prepared by heating 2:5-di-(1:4-diamino-2-anthraquinonyl)-1:3:4-oxadiazole with benzoyl chloride in nitrobenzene containing pyridine at 130°C. for 6 hr. The oxadiazole is made by condensing 2 mol. of 1-amino-4-nitroanthraquinone-2-carboxylic chloride with 1 mol. of hydrazine, cyclising with $SOCl_2$, and finally reducing the NO_2 groups with Na_2S .

R. K. F.

Bisindhenzopyrenequinone Sulphide—Vat Dye. General Aniline. USP 2,562,872

Benzanthrone-3-carboxylic chloride is condensed with diphenylene sulphide by heating at 75–80°C. for 3 hr. in nitrobenzene containing $AlCl_3$. After separating, the product is cyclised by heating for 1 hr. at 145–160°C. in a molten mixture of $AlCl_3$ and $NaCl$ through which oxygen is passed. The resulting orange vat dye—

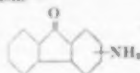


is fast to chlorine.

R. K. F.

1-Amino-4-fluorenylaminoanthraquinone-sulphonic Acid—Dye for Wool and Superpolyamides. General Aniline. USP 2,564,654

An aminofluorenone—

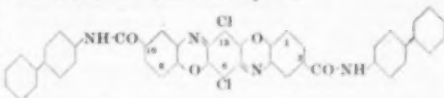


is condensed with 1-amino-4-bromoanthraquinone-2-sulphonic acid to give, as an alkali metal or NH_4 salt, a sparingly soluble dye which, from neutral dispersion, dyes nylon and wool level blues, fast to light, washing, and rubbing. Thus sodium 1-amino-4-bromoanthraquinone-2-sulphonic acid is refluxed in aq. ethanol for 6 hr. with 2-amino-fluorenone in presence of Na_2CO_3 , $NaHCO_3$, and $CuCl_2$.

R. K. F.

Triphenldioxazine Sulphur Dyes. DuP. USP 2,564,380

The dioxazine derivatives of USP 2,504,153 (2,5-d.p.c., 67, 88 (1951)) are modified, before sulphurising to red sulphur dyes, by introducing as substituents two aromatic amide radicals. Thus the compound—



made by first condensing 4-aminodiphenyl with 3-nitro-4-methoxybenzoic acid in presence of $SOCl_2$ and treating the resulting amide with chloranil, is heated at 75–80°C. for 30 min. with an $AlCl_3 \cdot 8C_2Cl_2$ complex.

USP 2,564,381

Instead of sulphurising the dioxazine derivative as described above, the amine initially used carries a thiocyanato group, the triphenldioxazinebisthiocyanate subsequently obtained being a red sulphur dye. Alternatively, a triphenldioxazine-2,9-dicarboxylic acid is condensed with 2 mol. of a thiocyanatoaniline. Thus 6:13-dichlorotriphenldioxazine-2:9-dicarboxylic acid is refluxed for 2 hr. with $SOCl_2$ in o - $C_6H_4Cl_2$, p -thiocyanatoaniline and pyridine are added, and heating is continued for 1 hr. at 120–130°C.

R. K. F.

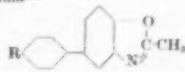
Halogenated Phthalocyaninedisulphonic Acids—Cotton Dyes. Gy. BP 676,542

Copper or nickel phthalocyaninedisulphonic acid is halogenated until 2–7 halogen atoms per mol. have been introduced to produce blue direct cotton dyes of better light and water fastness and improved affinity when compared with the unhalogenated compound. Thus chlorine is passed into sodium copper phthalocyaninedisulphonate in a melt of $AlCl_3$ and $NaCl$ at 170–175°C., and the product separated by pouring into dil. HCl .

R. K. F.

Mono- and Poly-methine Dyes. Gevaert. BP 675,654

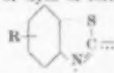
Mono- and poly-methine dyes containing at least one nucleus of formula—



(R = substit. phenyl) can be used as photographic sensitizers.

BP 675,656

The same is true of dyes of formula—



C. O. C.

Disperse-type Dye Powders. Celanese Corp. of America. BP 676,721

Dye powders with good dispersing properties are prepared by spray-drying an aqueous milled water-insoluble dye in presence of ligninsulphonic acid and a water-soluble alkali-metal salt. Thus, all parts being by weight, the dye (20) formed by coupling 2-bromo-6-chloro-4-nitroaniline with α -chloro-N,N-bis- β -hydroxyethylamine is milled for 48 hr with sodium ligninsulphonate (10), potassium pyrophosphate (10), and water (60), and spray-dried to a moisture content of 4-7%.

R. K. E.

Vat Dye Powders for Printing. Ciba. BP 675,800

Powders readily formed into printing pastes are obtained by adding not > 10% (on the dry wt. of dye) of a viscous water-soluble ether-carboxylic acid or ether-sulphonic acid (carboxyalkyl or sulphonyl derivative) of a high-polymer carbohydrate to an aqueous dispersion of a finely divided vat dye and drying the resulting paste.

C. O. C.

Flavonol Derivatives. Laboratoire Dausse.

BP 677,493

The tertiary-aminosulfonyl ethers of flavonol glycosides, e.g. rutin β -diethylaminoethyl ether, are much more soluble in water than the parent compounds.

C. O. C.

Carbon Black. Columbian Carbon Co. BP 675,819

Modification of BP 670,863 (U.S.D.C., 65, 275 (July 1952)), the make gas being more efficiently and more rapidly heated so as to promote partial pyrolysis of the gas.

C. O. C.

Carbon Black. United Carbon Co. USP 2,590,660

A method of obtaining increased yield of furnace black.

C. O. C.

Improving the Consistency, Reflectivity, and Stability, and Increasing the Fineness of Metallic Pigments. Aluminum Co. of America.

BP 676,641-2

The particle size of metallic paste pigments is reduced without increasing their covering area by blade-agitating a mixture of 70-85% metal flakes, not > 0.5% lubricant, and 15-30-29.5% mineral spirits, to commingle and subject the individual flakes to surface rubbing and shearing action on and between themselves. Agitation lasts for 1-12 hr. at not > 110°F.

C. O. C.

Bright Red Fire-resistant Selenium Cadmium Pigments. Deutsche Gold- und Silber-Scheideanstalt vormals Roessler. BP 676,921

Addition of vanadium compounds (6-5-3-0% by wt. calc. as vanadium pentoxide) to the raw mix for cadmium sulphoselenide pigments results in products which are much less sensitive to discoloration during production and use.

C. O. C.

Titanium Dioxide. New Jersey Zinc Co.

USP 2,589,909-10

Efficient production of high-quality pigment from titaniferous slags (of valency < 4), obtained by the smelting of non-titanium ores, in described.

C. O. C.

Dye-Gelatin Phosphors (IX p. 416).

Spectral Sensitivity of Light-fastness Standards. (XIV p. 420).

V—PAINTS; ENAMELS; INKS

Electrophoresis of Pigments in Organic Solvents—

II. H. Brintzinger, R. Haug, and G. Sachs. *Farbe und Lack*, 58, 143-150 (1952); *Chem. Abn.*, 46, 6401 (10 July 1952).

A detailed review on this subject (79 references) is followed by an account of work in which gas black, TiO_2 , and ZnS , suspended in mineral spirits, benzene, CCl_4 , etc., conducted electricity after application of a field of constant voltage, with the current rising gradually, but not continuously, to a maximum. This was caused by formation of chains which lasted a while after the field had been removed. Increase in voltage caused the current to rise and the resistance to fall. Pigments (TiO_2 , ZnO , chrome yellow, etc.) suspended in non-polar or weakly polar solvents were subjected to a field of 20,000 v. Some inorganic pigments (Pb_3O_4 , CdS) did not dance or form chains below 1000 v., but did so increasingly up to 5000 v.; above this voltage, bridges or chains were formed, the pigments

became immobile, and a fairly firm deposit formed on both electrodes. In an A.C. field above 1000 v. at a frequency of 50 hertz, pigments such as TiO_2 , ZnS , chrome yellow, and Milori blue formed threadlike growths from the lower electrode upward, until at 3000-4000 v. the tree-like growths had formed bridges and chains between the electrodes. The mutual attraction of the particles seems to be caused by their external polarisation, owing to increasing displacement of the diffuse ionic layer where the potential drop is great; near the electrode the ionic layer may even be partly lost.

C. O. C.

Cellulose Lacquers. XXIII—Effect of Pigments on

the Hardness of Coatings. Y. Inoue. *J. Soc. Textile Cellulose Ind. (Japan)*, 6, 147-150 (1959); *Chem. Abn.*, 46, 6403 (10 July 1952).

The indentation hardness of films of cellulose derivatives and acrylic polymers with and without plasticisers or other resins increased when pigments were added. The pendulum hardness increased solely with the amount of pigment, while scratch hardness markedly increased independently of the hardness of the unpigmented film. A relatively higher resistance of softer films to scratching here seemed due to the wider region of the stress distribution and higher elastic deformation.

C. O. C.

Stencils and Duplicating Inks. A. H. Woodhead.

Paint, 21, 283-286, 313 (Aug. 1952).

An account of the manufacture of typewriter stencils and of oil-based paste and liquid duplicating inks. Both types of ink are essentially carbon black in oil, and both can be water-in-oil emulsions stabilised largely if not entirely by the finely dispersed carbon black which is used almost exclusively. Pigments and toners used must be entirely free from any tendency to bleed in the medium.

C. O. C.

PATENTS

Printing Inks from Tall-oil-modified Alkyd Resins.

American Cyanamid Co. USP 2,590,654

Tall-oil-modified alkyd resins in an inert solvent are excellent vehicles for printing inks, are adaptable for use with a great variety of pigments, and particularly show excellent wetting properties when used with carbon black. Inks in which these resins are the vehicles enable printing to proceed much faster than ordinarily possible and dry very quickly.

C. O. C.

Bleachable Transfer Ink. H. J. Steiner.

USP 2,589,306

A water-permeable, bleachable ink for typewriter ribbons, carbon papers, etc. comprises a wax, not < 10% of a water-dispersible wetting agent, and a bleachable dye. If ammonium vanadate is incorporated in the ink, then imprints made by it can be chemically treated to render them legible even after they have been bleached.

C. O. C.

Stencil Duplicating Inks. A. B. Dick Co. BP 676,373

An aqueous composition of a dye or pigment, a cellulose ether or ester containing water-solubilising OH groups, and an aldehyde capable of insolubilising the cellulose derivative upon drying is a quick-drying ink which distributes itself quickly and evenly through inkpads and through the stencil.

C. O. C.

Paints containing Slaked Lime as Pigment.

Lord Mayor, Aldermen, and Citizens of Bradford.

BP 676,532

A water paint or colour wash is produced by mixing very hot freshly slaked lime with a crude soap obtained by saponifying wool grease with anhydrous NaOH . The product is suitable as a basis for distempers, etc.

C. O. C.

Translucent Coatings of Metallic Appearance. DuP.

BP 676,696

A coating composition, which yields a tough durable finish having a metallic appearance of great depth and brilliance, consists of a vehicle and a pigment, the latter being made up of—(a) prime pigment consisting predominantly of hydrous iron oxide, chromium tetrahydroxide, or lightfast Litholom nickel azo salt; (b) a pigment complementary in colour to the prime pigment in amount to yield a grey when mixed separately therewith; (c) at least one coloured tinting pigment; and (d) finely divided aluminium flake. If desired the "neutralising" pigment(s) can be selected in such a way that it (or one of them) can be used in excess of the required "neutralising" quantity to produce the desired colour. The value or lightness is adjusted by the amount of aluminium flake

added. This also enables manufacture of durable grey lacquers without the use of black or white pigments, or the manufacture of a pigmented composition of a specific hue using a pigment of that hue or an equivalent blend of more than one pigment in only minute proportions with respect to the total pigment present. Additionally it enables use of the desirable properties of hydrous iron oxide pigments in enamels and lacquers having better durability and a wider variety of hues than heretofore available. C. O. C.

Pigmented Coating Compositions. British Thomson-Houston Co. BP 675,936

Addition to pigmented resinous coating compositions of 0.00001–0.01% by weight (on the resin and pigment) of a liquid alkylpolysiloxane decreases the tendency for pigment flotation or for the composition to show "flocking" or "flow lines". C. O. C.

Glossy Emulsion Coating Composition. Lewis Berger & Sons. BP 676,420

An aqueous dispersion of a pigment, preferably of particle size 0.2–0.5 μ , which has been rendered "oleophilic" before or after dispersion, is mixed with at least part of the oil vehicle which is to form the discontinuous phase of the final emulsion. The pigment-in-oil-water-in-oil system so formed is then converted into a pigment-in-oil-in-water emulsion of use as a glossy enamel. C. O. C.

Enamelled Drying-oil Coatings. Lewis Berger & Sons. BP 665,496

Wrinkling, frosting, and gas-checking of films of drying oils or drying-oil-modified varnishes is prevented by heating the oil at 470–525° F., if desired in presence of a solvent, with 0.005–0.1% of an organic disulphide, e.g. diphenyl disulphide. E. C.

Metal Carboxylates—Driers. British Industrial Solvents. BP 677,175

Pb, Co, and Mn 2:4:4-trimethylvalerates when powdered yield free-flowing products, soluble in hydrocarbons. They are used as driers. C. O. C.

Metallic Powders and Pastes as Pigments (IV p. 406). Modern Pigments for Automotive Finishes (IV p. 406).

VI—FIBRES; YARNS; FABRICS

The St. Gallen Textile Industry. G. Thüner. *Textil-Rund.*, 7, 259–267 (June 1952).

A historical and pictorial record of the textile industry in St. Gallen with special reference to linen. 32 references. B. K.

High-contrast (Wide-field) Electron Microscope Objective and Fibre Replicas. V. F. Morehead.

Textile Research J., 22, 379–384 (June 1952).

The high-contrast, low-magnification lens designed by Hillier for ultra-thin sections is shown to be applicable for fibre surface replicas, and in many cases metal shadowing is found to be unnecessary. Three replicating techniques have been used successfully—viz. cellulose nitrate; evaporation; ethyl cellulose, heat and pressure moulding; and polystyrene, heat and pressure moulding. A. B.

Action of Light on Textile Fibres. H.-J. Henk. *Melland Textilber.*, 33, 488–491 (June 1952).

The effects of exposure to light on the physical and chemical properties of the common textile fibres, in their natural state or after various finishing or dyeing treatments, are reviewed, and explanations that have been advanced for the observed behaviour are given. A. E. S.

Theory of the Photolysis of Textile Fibres. A. Sippel. *Melland Textilber.*, 33, 645–649 (July 1952).

In a theoretical treatment of the photolysis of fibres in its relation to changes in degree of polymerisation (D.P.) and tensile strength, various assumptions are made concerning the fraction of the bonds linking monomer residues that are photosensitive and the fraction of these bonds that are significant in determining the tensile strength. Five possible cases are considered, and in each case a relationship is derived between D.P. and tensile strength. Only one of these treatments gives a relationship close to that found experimentally, and the assumptions made in this treatment are that a certain fraction of the linkages are both sensitive to photolysis and of significance in determining the tensile strength; that the absorption of a quantum can occur at any linkage; and that the energy

of photons absorbed at linkages that cannot undergo photolysis is transferred to linkages that can. This hypothesis leads also to the prediction of a quantum yield approaching unity for the photolysis reaction. This prediction is confirmed by experiments in which cellulose acetate films are given a known dosage of ultraviolet radiation (2537 Å.) and measurements made of the changes in D.P. It is suggested from theoretical considerations that it should be possible to improve the resistance of textiles to irradiation by treatment with suitable substances, even if these are introduced only into the so-called amorphous regions of the fibre. A. E. S.

Action of Light on Vat-dyed Cellulose. C. B. Kothari and G. M. Nabar. *J.S.D.C.*, 68, 66–67 (Feb. 1952).

Constitution and Fine Structure of Natural Cellulose. K. H. Meyer. *Melland Textilber.*, 33, 485–488 (June 1952).

A survey is made of present knowledge of the constitution and fine structure of natural cellulose. It is emphasised that the true degree of polymerisation of natural cellulose is much greater than that indicated by conventional viscosity measurements and that molecular chains may, in fact, extend to the full length of the cell. X-Ray analysis does not enable the fine structure to be established with certainty, but it is highly probable that the scheme of Meyer and Misch (see *J.S.D.C.*, 53, 259 (June 1937)), in which adjacent chains are mutually parallel, but oriented in opposite directions, is essentially correct. The chains lie parallel to the fibre (fibril) axis, and although the crystalline arrangement is not perfect, it is incorrect to refer to "crystalline" and "amorphous" regions and to attach fundamental importance to calculations of the crystalline : amorphous ratio. Diffuse scattering of X-rays is to be attributed not only to the presence of some regions of poorer orientation, but also to the small size of the crystallites and to the presence of foreign amorphous matter. The results of the X-ray examination of rolled films of natural cellulose from tunicates (tunicin) are of interest. There is a crystal plane parallel to the film surface, the chains being grouped in an array that is parallel with respect not only to the direction of the long molecular axis, but also to a direction perpendicular to this. The X-ray evidence, supported by work based on double-refraction and adsorption measurements, led the author in 1939 to the conclusion that native cellulose consists of individual fibrils, not anastomotically connected, and that there are intervening spaces, which may contain air, water, or amorphous cell substance. Estimates from adsorption experiments of the inner surface of the fibres (available surface of the fibrils) led to a value of 200 Å. for the diameter of a fibril. Recent electron-microscopic work fully supports these conclusions. It shows that primary cellulose, from the wall of a young, growing cell, consists of a loose felt of fibrils of the expected size, and that fibrils of the same size form the basis of bacterial and animal cellulose and of the secondary cellulose of natural fibres, in which they adhere together in the form of parallel bundles. A secondary layer of cellulose from the wall of a fibre is represented by the author as having a highly crystalline structure, similar to that found in tunicin films; i.e. not only have we mutually parallel fibrils, each containing mutually parallel chain molecules, but the planes of the glucose residues in the molecules of one fibril are mutually parallel and are substantially parallel also to those in the next fibril. Tunicin is discussed in some detail. Electron micrographs show the presence of some finer fibrils (≈ 100 Å.), or the fibrils may be flat, as postulated by the author on evidence obtained from rolled films. Examination of acid-treated tunicin shows that the fibrils are attacked superficially and become brittle; this effect is considered to be associated with the increase that occurs in the sharpness of the X-ray pattern of natural cellulose when the latter is treated with acids, and it is suggested that the inside of a fibril is more perfectly crystalline than the outside. There are five reproductions of electron-micrographs and 24 references. A. E. S.

Partial Acetylation of Cotton. A. B. Cooper, R. T. Voorhes, E. M. Buras, and C. F. Goldthwait. *Textile Industries*, 116, (1), 97–102, 194–195 (1952); *Amer. Dyestuff Rep.*, 41, 436 (7 July 1952).

Cotton is purified by removing non-cellulosic material,

activated by soaking not < 4 hr. at room temp. in glacial acetic acid, squeezed and drained, acetylated with a cooled mixture of commercial glacial acetic acid, acetic anhydride, and perchloric acid catalyst, and washed with cold, and finally hot, water. Degree of acetylation is determined by saponification with NaOH followed by back-titration; dyeing tests assess the uniformity of acetylation. Moderate coats make this a commercial proposition in view of the great increase in length of life of the material when used for certain purposes, e.g. heat-resistant pad covers for steam presses, zeolite bags for water softeners, and fishing twines. J. W. B.

Pyrolysis and Combustion of Cellulose in the presence of Inorganic Salts. K. Tamaru. *Bull. Chem. Soc. Japan*, **24**, 290 (Dec. 1951).

Correction to previous paper, *ibid.*, **24**, 167 (Sept. 1951); J.S.D.C., **68**, 133 (April 1952).

Cellulose Studies. XVII—Heterogeneous Acid Degradation of Cellulose. XVIII—Homogeneous Degradation of Cellulose in Phosphoric Acid. P. W. Bauer and E. Paces. *Textile Research J.*, **22**, 385-397, 397-404 (June 1952).

XVII— Acid degradation of cellulose normally results in a rapid drop in the degree of polymerisation followed by a much slower decrease at degrees of polymerisation of about 200. A series of experiments carried out for the purpose of determining the actual mechanism of the reaction is described, including mild acid hydrolysis of mercerised cotton cellulose, of 2500 years old mummy cloth, and of cotton bolls picked at various intervals after flowering.

XVIII— It has been found that a solution of cellulose in 85% phosphoric acid allowed to react at 21°C. for 19 months still contains, in 40% yield, a cellulose fraction with a degree of polymerisation > 100, indicating some error in the determination of the published rate constants for this hydrolysis. A crystalline glucose was isolated from the phosphoric acid degradation of cellulose. A. B.

Plastic Deformation of Regenerated Cellulose Fibres. I—Dichroic Study of the Deformation of freshly Prepared Fibres. S. Okajima and Y. Kobayashi. *Bull. Chem. Soc. Japan*, **24**, 289 (Dec. 1951).

Correction to previous paper, *ibid.*, **24**, 88, 89 (June 1951); J.S.D.C., **67**, 471 (Nov. 1951).

II—Deformation of freshly Prepared Fibres from the Standpoint of Refractive Indices. S. Okajima and S. Hayama. *Loc. cit.*

Correction to previous paper, *ibid.*, **24**, 91 (June 1951); J.S.D.C., **67**, 471 (Nov. 1951).

Acetyl Groups in Jute Fibre. P. B. Sarkar. *J. Textile Inst.*, **43**, T 290 (June 1952).

In reply to Bhattacharjee and Callow (*ibid.*, **43**, T 53 (1952)) it is stressed that the presence of acetyl groups in jute fibre has been long established, and that by estimating acetic acid from raw and ClO₂-delignified jute it was shown that lignin contains no acetyl groups. Recently, crystals of ammonium acetate have been obtained from the ammonia extract of defatted jute. It is considered possible that acetyl groups are linked to hexosans. J. W. B.

Fungal Decomposition of Jute Fibre and Cellulose. III—Decomposition of Cellulose as Influenced by its Physical State and by Associated Substances. S. N. Basu and S. N. Ghose. *J. Textile Inst.*, **43**, T 278-T 289 (June 1952).

Using a large number of jute-decomposing fungi, it is shown that the lignin has a strong protective action on the remainder of the fibres. Lignin-free jute is more susceptible than cotton or jute α-cellulose, owing to the presence of hemicellulose, which stimulates cellulose decomposition by way of a cellulose-splitting enzyme which may in some degree be specific for the fungus. Further stimulation is provided by micronutrients in the water-soluble matter in jute. Increased surface area and low crystallinity: amorphous ratio favour cellulose decomposition, but decrease in chain length has no effect; in fact, hydro-cellulose is more resistant, possibly because of increased crystallinity. Fungi are classified into six groups according to their severity of attack, ranging from those which attack in presence of lignin to those having no action on hemicellulose or associated cellulose. J. W. B.

Electron-microscope Study of the Wool Fibre Cuticle. J. Ames. *J. Textile Inst.*, **43**, T 262-T 267 (June 1952).

Merino wool sections, approx. 0.2 μ. thick, are cut in a rocking microtome, the technique being fully described. Electron micrographs (× 10,000) include normal and oblique sections of untreated and chlorinated fibres. Some cuticle structure is visible, and three layers are seen which may, despite anomalies, be the epi-, exo-, and endo-cuticles. No definite evidence is, however, obtained of the nature of the fibre histology between cuticle and cortex. J. W. B.

Deposition of Polymers on the Wool Fibre and Allwörden's Reaction. O. E. Ford. *Melland Textiler*, **33**, 597 (July 1952).

When a wool fibre upon which a polymer has been deposited, e.g. by treatment with N-carboxyglycine anhydride (see J.S.D.C., **62**, 4 (Jan. 1946)), is subjected to Allwörden's reaction, the bubbles formed attain a much larger size than usual before bursting. It is considered that the polymer reinforces the epicuticle, so that a greater pressure is required to burst the bubble membrane. A similar effect results from the treatment of wool with Sn salts. The results confirm that the Allwörden bubble membrane is identical or closely associated with the epicuticle. A. E. S.

Molecular Size of Proteins from several Wools solubilised in Aqueous Urea. W. H. Ward. *Textile Research J.*, **22**, 405-415 (June 1952).

Wools solubilised in concentrated aqueous urea with mercaptoethanol are compared by molecular kinetic measurements to show possible variations characteristic of fineness, breed, or country of origin, and it is shown that no significant differences appear in the molecular size or shape of the protein. The average molecular weight of the dispersed protein, amounting to about 65% of whole wool, is found to be 14,000. A. B.

Structural Parameters of the Micellar Lattice of High-polymer Fibres. R. Hosemann. *Kolloid-Z.*, **125**, 149-156 (March 1952).

The discontinuous distribution of small-angle X-ray intensities in photographs of α- and β-keratin, collagen, polyurethane, and triethyl cellulose fibres is analysed by a mathematical statistical method without reference to any preconceived structural model. This analysis unambiguously indicates that the fibres possess a micellar lattice whose dimensions are—α-keratin 198 Å × 83 Å; β-keratin 95 Å × 34 Å; collagen 655 Å × 10 Å; polyurethane 70 Å × 10 Å—the long spacing being parallel to the fibre axis and the short one perpendicular to the axis but in the side-chain direction. The micelle lattice of triethyl cellulose appears to indicate a "solid of gaseous type". The diffuse nature of the diffraction arcs is shown to be due to the ends of the micelles not exactly coinciding (dislocations) and to the surfaces varying in orientation. The dislocations of the long spacing are less than 1% in the proteins but > 16% for polyurethane; in the other direction they are only 4-5% for keratin but 15% for collagen. The angular dispersion of the micelle length in the proteins is small (< 1%) but that of the width is 2° for β-keratin, 7° for collagen, and 20° for α-keratin. The values for polyurethane are larger in all cases. L. P.

Ultraviolet Absorption of Silk Fibroin—I. E. Schauenstein. *Melland Textiler*, **33**, 591-597 (July 1952).

A review is given (35 references) of work by the author and others on the examination of silk fibroin by ultraviolet absorption methods. In addition to absorption due to electronic transitions, which is associated with the presence in the molecule of various chromophoric groupings, there is Tyndall absorption, which is associated with the shapes and sizes of the particles present and is expressed by the Rayleigh equation $k = a\tau^n$ (k = extinction coeff. at wave number τ ; and a and n are constants determined by the dimensions of the particles; $n = 4$ for particles whose greatest dimension is < 0.1 of the wavelength of the light, and $n < 4$ for larger particles). For pure Tyndall absorption, a double-logarithmic plot of k against τ gives a straight line, whose slope ($= n$) and position give some indication of the sizes of the particles. At one extreme there are gelatin soln. and a soln. of native fibroin in 8% NaOH, which give closely situated curves for which $n = 4$.

and k is low; at the other there is a film of native fibroin ($n = 1.2$, and k is high); a soln. (pH 8) of silk fibroin renatured according to Coleman and Howitt (*J.S.D.C.*, **64**, 121 (March 1948)) is intermediate in behaviour and gives a curve close to that of the globular component of actomyosin, which has an estimated mol. wt. of 70,000 and, like renatured fibroin, can be transformed reversibly into the fibrous form. Absorption due to electronic transitions is readily separated from that due to the Tyndall effect. Application of absorption measurements to the estimation of the tyrosine content of fibroin is discussed, and the spectra obtained from solutions and films of renatured fibroin and from films prepared from native silk gel are compared with the spectra of tyrosine and glycyltyrosine anhydride (a diketopiperazine deriv.). It is thus shown that tyrosine is combined in the anhydride form in all these preparations (see *J.S.D.C.*, **66**, 158 (Feb. 1950)). The enol form of one $-CO-NH-$ of the diketopiperazine residue gives rise to a band at 3950 mm^{-1} , which can be observed also in the spectrum of glycyltyrosine anhydride; after vigorous rolling and stretching treatments, films of native silk show increased long-wave absorption, attributable to the enol form of the remaining $-CO-NH-$. It is considered, therefore, that the formation of diketopiperazine during the breakdown of fibroin is due not to secondary reactions, as considered by a number of authors, but to the presence of the diketopiperazine residue in the fibroin molecule. X-Ray data are highly consistent with this hypothesis. Recent work on renatured fibroin (Signer and Glanzmann: cf. *J.S.D.C.*, **65**, 133 (April 1952)) has indicated that the renaturing process (dissolution in cupriethylenediamine soln., followed by neutralising and dialysing the soln.) leads to degradation of the fibroin. It is considered, however, that the spectrum data and also X-ray, optical, and mechanical measurements on films show, in agreement with the views of Coleman and Howitt, that fibroin does not undergo appreciable degradation in this process. The globular and fibrous states of renatured fibroin are regarded as substantially identical with native fibroinogen gel in the silk gland and with fibrous cocoon silk respectively, but films formed from renatured fibroin and subjected to rolling and stretching are not identical in the finer details of structure with films prepared from native silk.

A. E. S.

Action of Peracetic Acid on Rabbit Hair. H. G. Fröhlich and B. Hauptmann. *Deutsche Textilgeserbe*, **54**, 27-28, 30-32 (1952); *Chem. Abs.*, **46**, 6389 (10 July 1952).

Oxidation of cystine in wool and rabbit hair by 1-6% peracetic acid proceeds rapidly in the first 2 hr., slowing down later so that after 2 hr. 75% and after 18 hr. 92% of the cystine was converted into cysteic acid. The treated fibres showed increased alkali solubility and decreased capacity for acid binding, the extent of which was related to the degree of oxidation. Decreased acid binding was attributed to acetylation of the amino groups, and was not seen after treatment with an equivalent solution of H_2O_2 and acetic acid. Increase in swelling of the fibres was present only after 75% or more of the cystine was oxidized. Rabbit hair treated with peracetic acid for 2 hr. and then shaken with 10% ammonia overnight gave an insoluble residue composed of striated swollen membranes, similar to the intermediate membranes isolated from wool, and pigment granules surrounded by membranes. Hydrolysis of rabbit hair with 5 N-HCl for 2 hr. also gave a similar small insoluble residue.

C. O. C.

Structure-Property Relationships in Polyethylene Terephthalate Copolyesters. I—Melting Points. O. B. Edgar and E. Ellery. *J.C.S.*, 2633-2638 (July 1952).

The melting points of polyethylene terephthalate-adipate and polyethylene terephthalate-sebacate copolymers are measured by a penetrometer method (described in detail), and those containing > 40 mol. % of ethylene terephthalate units are found to be, for a given molar composition, approx. the same in the two series, the X-ray diffraction patterns of such polymers being essentially those of polyethylene terephthalate itself, but with increased amorphous scattering. Flory's relationship between the crystallite m.p. T_m and the "mole" fraction X of the units forming the crystallites is found to hold for values of X between 1.0 and 0.7, though for lower values of X the m.p. were lower than the calculated values. The

latent heat of fusion, calculated from the slope of the $\ln X - 1/T_m$ curve as $X \rightarrow 1$, is similar to that for the aliphatic polyesters, and indicates that the high m.p. of polyethylene terephthalate is due to chain rigidity, rather than to strong interchain attraction.

H. H. H.

Structure-Property Relationships in Polyethylene Terephthalate Copolyesters. II—Second-order Transition Temperatures. O. B. Edgar. *J.C.S.*, 2638-2643 (July 1952).

The penetrometer technique (cf. Part I, preceding abstract) when applied to quenched polymers affords a new method for the measurement of second-order transition temperatures. With copolymers of polyethylene terephthalate containing as the modifying component varying proportions of the polymethylenedicarboxylic acids, the second-order transition temp. is found to be a linear function of the number of p -phenylene groups per 100 aliphatic chain atoms. Anomalies are observed when the modifying component is either 1,2-propylene glycol or diphenyl-4,4'-dicarboxylic acid. It is suggested that the p -phenylene groups have little effect on interchain forces, but cause a marked increase in chain stiffness by reducing the possibilities of change of shape available to a given length of chain. This stiffening effect also accounts for the low entropy of crystallization and consequent high m.p. of polyethylene terephthalate.

H. H. H.

Stress Relaxation and Shrinkage in Fibres. M. C. Chen, T. Ree, and H. Eyring. *Textile Research J.*, **22**, 416-423 (June 1952).

The measurement of the stress relaxation with time at constant strain of Saran (polyvinylidene chloride) is described. The shrinkage of Saran at 67°C. is also measured, using the stress increase with time. A. B.

PATENTS

Viscose Rayon. H. A. Kuljian. *BP* 676,831
Production of free sulphur in the coagulating liquor is avoided by degassing the liquor and saturating it with an inert gas while protecting the surface of the liquor from contact with air or other gas containing free oxygen.

C. O. C.

Continuous Processing of Rayon Filaments. DuPont. *BP* 675,788

The filaments are continuously fed on to a rotating cylinder, and after having gone round this cylinder the requisite number of times are taken off to another cylinder or for storage. The filaments may be treated on each cylinder, e.g. treated with acid or dried. C. O. C.

Washing Viscose Fibres. Société de la Viscose Suisse. *BP* 674,869

Apparatus is described for washing viscose fibres in a series of wet treatments, the yarn passing through squeezing rollers at the end of each process so that the tension is held at a constant low value. W. G. C.

Plasticising Cellulose Acetate. C. Diamond. *BP* 674,883

Cellulose acetate is plasticised by treatment with an aqueous dispersion of a plasticiser, e.g. dimethyl phthalate, in presence of a cation-active agent, e.g. cetylpyridinium bromide. W. G. C.

Groundnut Protein Fibre. Courtaulds. *BP* 674,755

Regenerated protein fibres are made resistant to cold water by treating with an aqueous bath containing a halogenated aldehyde, e.g. chloral, and then drying, the process being carried out in the absence of formaldehyde.

W. G. C.

Fluid Polyamide Solutions. DuPont. *USP* 2,590,642

Polyamides whose recurring unit consists of α -amino acid units and the chain of $-N-CO-$ units yield fluid solutions in a solvent or solvents thereof, of energy density < 95, containing a small proportion of another organic compound or compounds soluble in or miscible with the solvent and of energy density > 95. Thus a solution of improved fluidity is obtained of the linear copolyamide (degree of polymerisation > 24) of L-leucine and DL- β -phenylalanine by use of 5% m -cresol in benzene.

C. O. C.

Polyester Fibres. Wingfoot Corp. *BP* 665,272

Polyesters suitable for preparing fibres of high strength are obtained by condensing a glycol and a diacid and, if desired, a mercapto-alcohol, in which, in each case, the two functional groups are separated by > 3 atoms, with

approx. the stoichiometric amount of a dibasic acid chloride in which the two acyl groups are attached to different carbon atoms.

BP 665,271

Terephthaloyl chloride, tetramethylene glycol, and a polymethylsulfonethiol of 4-10 C are used to produce the polyesters.

E. C.

Readily Dyable Acrylonitrile Polymers. Industrial Rayon Corp.

BP 676,889

Copolymers of acrylonitrile and monovinyl ethers of aminoalcohols or salts thereof are readily dyed with acid dyes and formed into molecularly oriented filaments.

C. O. C.

Polysulphone Triazoles or Polysulphone Amino-triazoles. BrC.

BP 676,785

Those polymers containing sulphone groupings between successive triazole or aminotriazole rings, when of sufficiently high mol. wt., are suitable for melt spinning into fibres.

C. O. C.

Reducing the Diameter of Non-metallic Continuous Fibres. International Co. for Industrial Exploitation and A. B. Molino.

BP 677,016

The fibres are passed through a liquor which attacks them physically or chemically, a flow of gas bubbles being simultaneously directed into the fibres, and the bubbles being caused to separate from the fibres at regular periods of time; e.g. hydrogen is formed by electrolysis below the fibres, separation of the bubbles from the fibres being effected by periodic impulses generated by sound or ultrasonic waves. The process is particularly applicable to glass fibres using dil. HF as the liquid.

C. O. C.

Colloid Science—A Chapter in Chemistry (VIII p. 413).

VII—DESIZING; SCOURING; CARBONISING; BLEACHING

Enzymatic Desizing of Fabrics. W. Julicher, J. Voss. *Melland Textiles*, 33, 511-516; 516-517 (June 1952).

Julicher gives a review of the properties and application in desizing of the three main types of amylase. Classification of these as α - and β -amylases is of no technical value, for it leads to the assumption that products, e.g. bacterial and pancreatic amylases, that are fundamentally distinct in properties are identical. The older method of classification as saccharifying and liquefying amylases is much more satisfactory. These classes are distinguished particularly in respect of temp. sensitivity—saccharifying enzymes are easily destroyed by heat, while liquefying enzymes are much more stable, and attain adequate activity only at high temp. Pancreatic amylases are strongly saccharifying. They give very rapid desizing under optimum conditions (55°C., pH 6.6), but their stability and activity are very sensitive to changes in pH and temp. Their thermal stability is higher in presence of Ca ions, and desizing by these enzymes, and also by bacterial amylases, should be done in hard water. Modern, stabilised pancreatic preparations are stable for a long time under the recommended desizing conditions, and, provided that these conditions can be carefully controlled, they give rapid, complete desizing. Bacterial enzymes are strongly liquefying and weakly saccharifying. They must be used at high temp. ($\sim 70^\circ\text{C}$.) and are not greatly sensitive to variation in pH, the optimum value being ~ 7 . They do not readily give complete desizing, but the result is often adequate. Commercial brands of bacterial amylase vary greatly in thermal stability. Malt amylases are strongly saccharifying and strongly liquefying. On heating for a short time at 70°C ., saccharifying power is lost, but liquefying power is retained; they may be used for desizing at this temp., the optimum pH being ~ 7 , but they are normally used at 55-60°C. and at pH 4.8-5.4. Their action is slow, but not greatly sensitive to fall in temp., and they are very suitable for the most widely used method of desizing—by impregnating the fabric and allowing it to lie—particularly as acid liberated in fermentation caused by infecting micro-organisms does not greatly affect the activity of the amylase. Desizing must in all cases be preceded by the swelling of the starch, which may be a separate operation. At this stage, wetting agents are often used, and the possible inhibiting effect of such agents on the enzyme must be taken into account.

The iodine test is considered to give the best criterion of the completion of desizing. The results of Voss (see J.S.D.C., 68, 58 (Jan. 1952)), which are not in accord with some of Julicher's assertions, are criticised. These results indicate that fall in activity with fall in temp. is less for bacterial amylase than for malt amylase, which is contrary to experience. The experiments, however, bear little relation to practical conditions—they were done on dissolved starch, not on swollen material, and the pH was not adjusted according to the requirements of the different enzymes. Also, the method of estimating the degree of starch breakdown by precipitation with methanol is criticised. The details given by Voss are not sufficient to enable the experiments to be repeated exactly, but some experiments are now done along the same lines, using, however, the appropriate pH for each enzyme. The results obtained are quite different from those of Voss, and it is shown that there is no simple relation between the amounts of material precipitated by methanol from the treated soln. and the readiness with which solid material obtained by evaporating the soln. redissolves in water. The method is therefore of no practical significance.

In reply to Julicher's criticism, Voss states that his procedure was dictated by the absence of a satisfactory method of determining residual starch on fabric. Details of his method are given; the concentrations of the reactants were chosen from a consideration of the conditions pertaining in practical desizing. The concn. of enzyme used by Julicher was far too high and would account for the differences in his results. The cause of the anomalous behaviour of malt amylase under the experimental conditions used has been elucidated in further work, which is to be published. It is pointed out that the end-point in technical desizing is not always indicated by a negative iodine test—the degree and type of desizing necessary depend on the subsequent treatment that the goods are to have; thus, if they are to be kivered and bleached, then a treatment that leaves a residue of soluble dextrins, which gives a violet iodine coloration, is quite satisfactory.

A. E. S.

Desorption of a Direct Cotton Dye from Cellulosic Fibres. J. H. E. Jackson and H. A. Turner. *J.S.D.C.*, 68, 345-352 (Sept. 1952).

Studies on Monomolecular Films. VII—Effects of Metallic Ions on the Monomolecular Film of Stearic Acid. T. Sasaki and R. Matsuura. *Bull. Chem. Soc. Japan*, 24, 274-278 (Dec. 1951). IX—**Effect of Dissolved Dyes on the Monolayer of Stearic Acid.** R. Matsuura. *Ibid.*, 24, 282-285 (Dec. 1951).

VII—The effects of various metallic ions on the pressure-area curve of monomolecular layers of stearic acid spread on the surface of the soln. may be classified into two groups—(a) those making the film condense, e.g. Ca, Ba, Mg; (b) those making the film expand, e.g. Th, Al, Fe, Co, Cu, Zn, Hg, Ni. This is explained in terms of the molecular structure of the soaps formed. It is suggested that the soaps of group (b) metals have a complicated polymeric structure.

IX—The effect of dissolved dyes on a monolayer of stearic acid on the surface of the soln. has been studied. A considerable expansion of the layer occurs with soln. of Phloxine and Congo Red, which is explained in terms of van der Waals adsorption. It is considered that the expansion caused by basic dyes is due to ionic interaction, which may be analogous to the formation of soaps of group (b) metals (Part VII).

A. J.

Nature of Built-up Film. I—Adsorption of Ions on Built-up Film of Stearic Acid. II—Deposition of Monolayer of Stearic Acid from Surface of Water containing Copper, Aluminium, and Thorium Ions. M. Muramatsu and T. Sasaki. *Bull. Chem. Soc. Japan*, 25, 21-25, 25-28 (Feb. 1952).

I—Built-up layers of stearic acid are prepared by depositing on a stainless steel slide a multilayer of a mixture of stearic acid and barium stearate, treating with 0.01 N-HCl to give a monolayer of stearic acid, and depositing additional layers of stearic acid on this. The adsorption of barium, mercuric, cupric, aluminium, and thorium ions from aq. soln. at various pH is determined as the increase I_1 in thickness of the layers. Ba shows a continuously increasing adsorption with increasing pH.

while other ions have an optimum pH range for adsorption. Skeletonisation for 30 sec. in benzene gave considerable differences in thickness between adsorbed and untreated films, owing to resistance of metallic soaps to dissolution, and the number J_2 of stearic acid layers taking part in adsorption is obtained. The ratio J_2/J_1 gives the increase in thickness for conversion of a monolayer of stearic acid to a metallic soap. For Ba this is constant over the pH range 3-9. Similar experiments with films built up by successive transfer of monolayers from water show that Ba can only penetrate four layers at pH 7.4, and Al at pH 4.3.

II—Monolayers of stearic acid have been transferred from water containing Cu, Al, Th, and Ba ions on to solid surfaces. All these films are hydrophobic, with contact angles between $92 \pm 12^\circ$ for Ba and $110 \pm 6^\circ$ for Th. Conditioning the films in soln. of the metallic ions in some cases causes wettability. Tentative explanations are offered on the basis of a macromolecular structure for metallic soaps. A. J.

Physicochemical Properties of the Surface of Aqueous Solutions. II—Foam Stability and Mechanical Properties of the Surface. H. Kimzuka and T. Sasaki. *Bull. Chem. Soc. Japan*, 24, 230-234 (Nov. 1951).

Aqueous solutions of 31 substances are examined, and the following properties measured:—(a) Surface viscosity, using a viscometer consisting of a vertical glass thread carrying a horizontal wire at the lower end which dips into the surface. The wire is displaced a short distance x , and released after ageing, and the rate of return measured, when $l(dx/dt) + mx = 0$ (l = surface viscosity and m = const.). (b) Surface tension γ . (c) Membrane effect, expressed as difference between apparent surface tension for receding and advancing meniscus. (d) Foam height, produced after shaking under standard conditions. (e) Foam life t , the time for foam height to decrease by 50%. A close correlation is found between l and t , and for the system saponin-ethanol-water between l , t , and membrane effect. A. J.

Studies on Foams. VII—Theory of Foam Formation of Dye Solutions. M. Nakagaki. *Bull. Chem. Soc. Japan*, 24, 269-274 (Dec. 1951).

The energy E required to remove unit area of adsorption layer from the surface of a dye soln. is calculated from a consideration of the dynamic equilibrium of adsorption and desorption at the surface of the soln. and the area of the dye molecule. A comparison of E and foaming ability of soln. of triphenylmethane dyes shows good agreement. A max. in the foaming ability-concn. curve of Methyl Violet 5B is explained by its high surface activity, as the thickness of the adsorbed layer is of the same order as the range of molecular forces. A. J.

Soiling and Soil Retention in Textile Fibres—Cotton Fibre Grease-free Carbon Black Systems. J. Compton and W. J. Hart. *Ind. Eng. Chem.*, 44, 1401 (June 1952).

Correction to previous paper, *ibid.*, 43, 1564-1569 (July 1951); *J.S.D.C.*, 67, 475 (Nov. 1951).

Effects of Magnesium Ions on the Detergency of Wool using Sodium Dodecylsulphate. T. E. Jordan, G. Volz, J. Gelb, and C. Romanovsky. *Amer. Dyestuff Rep.*, 41, 413-414 (7 July 1952).

Mg ions are incorporated in a sodium dodecylsulphate-sodium sulphate mixture by partial neutralisation of the sulphonic acid with MgO , by adding $MgSO_4$ to neutralised slurry, by intimate dry blending, or by adding $MgSO_4$ to dilute detergent soln. prior to the wash test. Detergency tests are carried out on a soil-cloth preparation in a Launderometer, and show that Mg ions increase severalfold the detergency properties for wool, the optimum composition being approx. 40% organic content, 15% $MgSO_4$, and 45% Na_2SO_4 . J. W. B.

Chemical Changes occurring in Wool during Wet Processing. A. J. Farnworth. *Textile J. of Australia*, 27, 223-227 (April 1952).

After giving accepted formulae for the constitution of wool, some work is outlined on the effect of scouring, carbonising, and neutralisation of wool on the chemical

constitution of this fibre. Full details of this experimental work are to be given in a later paper. B. K.

Recent Developments in Bleaching with Hydrogen Peroxide. W. S. Wood and K. W. Richmond. *J.S.D.C.*, 68, 337-344 (Sept.); 397 (Oct. 1952).

Investigations on Fur Dressing and Finishing. VI—Effect on the Hair and Leather of Bleaching Natural-coloured Furs. F. Stather and Königfeld. *Gas. Abhandl. deutsch. Lederinst. Freiberg/8a.*, (7), 5-20 (1951); *Chem. Abs.*, 46, 5853 (25 June 1952).

Furs are usually bleached by alkaline killing, mordanting with $FeSO_4$, treating with H_2O_2 , and finally removing the iron with oxalic acid. Judging from the increased alkali solubility, digestibility of the hair by pancreatin and loss in tensile strength of both hair and leather of rabbit skins given $H-CHO$ and Leipzig pickle, all these processes cause some deterioration in both skin and hair. Deterioration due to killing and mordanting is slight. Concentration of $FeSO_4$ should not be > 10 g./litre. Addition $NaHSO_4$ to the mordant gives better bleaching and facilitates final removal of iron. Deterioration due to bleaching increases with time, temp., pH, and concn. of H_2O_2 , as does the bleaching action up to a maximum. Least deterioration with satisfactory bleaching is obtained with 30 g. H_2O_2 (30%) per litre at 30-35°C. and pH 8 for not > 5 hr. Starting bleaching at pH 5 for 1 hr. is advantageous. Addition of Aviol AH extra or sulphated castor oil protected neither the hair nor the skin but did give better bleaching. Other commercial protective agents were without effect. Addition of $H-CHO$ to the killing bath reduced the slight deterioration that occurs there. C. O. C.

PATENTS

Purification of Cellulosic Materials. Celanese Corp. of America. *BP* 676,910

The materials are chlorinated and then treated with 0.1-5% aq. caustic alkali at not $< 160^\circ$ C. C. O. C.

Removing Graphite from Nylon. American Textile Co. *BP* 674,580

Aqueous solutions of dispersions of hydroxides or carbonates of alkali and alkaline-earth metals (0.5-20.0% hydroxide or carbonate on the weight of the liquor) are highly effective in removing graphite from nylon. C. O. C.

VIII—DYEING

Dyers of Old Traunstein and their Craft. B. Brückner. *Melliand Textilber.*, 33, 671-672 (July 1952).

Quotations are given showing how the lists of dyers' materials and of various dyed and printed textiles that are to be found in the Traunstein Inventories (1520-1815) enable a picture to be formed of the dyer's craft and its development as a cottage industry. A. E. 8.

Quantitative Relationship between Depth of Dyeing and Light Fastness. J. C. Eaton, C. H. Giles, and M. Gordon. *J.S.D.C.*, 68, 394-396 (Oct. 1952).

Colloid Science—A Chapter in Chemistry. E. Rideal. *J.C.S.*, 2479-2487 (July 1952).

Review of the history of colloid science. It is in the correlation of thermodynamic with molecular concepts that problems arise, e.g. in the concept of the orientated monolayer, which was derived from the Gibbs equation. In a reference to emulsions (e.g. soaps), it is stated that their stability and the exact causes of the phenomenon of spontaneous emulsification are as yet not clearly understood, but it is noted that emulsion stability involves consideration of the stabilising influence of charge, surface viscosity, surface rigidity, and elasticity, as well as interfacial tension, and that quantitative methods of investigating these factors are now being considered. It is remarked, on the subject of ionic distribution around ionic crystals, that whilst lattice fit and charge are the two most important properties of the potential-determining ions for the ionic crystals, in the cases of fibres of organic materials such as wool and the synthetic polyamide fibres on the one hand, and of cellulose on the other, the situation is more complex. It seems clear that the $-NH_3^+$ and, to a certain extent, the $-CO-NH-$ groups are the ionic accepting groups for the dye anions in the former case, and the

CO_2 —and, to a certain extent, the CH_2OH groups (present in cotton) in the latter case, but a comparison of the dyeing properties of a homologous series of dyes indicates that van der Waals contributions from the rest of the molecule, and for cellulose actual adhesion along the cellulose chain, play an important part in the free-energy change on adsorption. Of the interesting problems which have arisen concerning macromolecular synthesis, the various approaches are noted which have been made to the synthesis of polypeptides, to the evaluation of the B and the A terms in the Arrhenius equations for each of the individual steps of the chain reactions involved in chain initiation, propagation, and termination, to the directive influence of groups in linking up asymmetrically substituted olefins in the head-to-tail or head-to-head position, and finally to the distribution of units in the chain when interpolymers are formed between two and even three differently substituted olefins. Attention is also drawn to improved methods for the determination of osmotic pressures of polymer solutions, and to the extension of the theory of elasticity of randomly coiled deformable networks to embrace the swelling and imbibition of solvent into deformable cross-linked structures. Finally, reference is made to the more detailed knowledge now available of the conditions which determine the relative ease with which macromolecular chains can adsorb with one another and fit into a crystal lattice; with increasing ease of adsorption, one passes successively from rubbers to plastics and thence to fibres.

H. H. H.

Methods of Solution of Axially Symmetric Diffusion.

A. Dobrowsky. *Kolloid Z.*, **126**, 1-14 (April 1952).
The solution, in terms of Bessel functions, of axially symmetric diffusion, e.g. into cylindrical bodies, is compared with a numerical method which treats the medium as a set of cells. The concentration in each cell at various times is calculated by considering the diffusion into and out of each cell. Pictures and details of construction are also given of a hydrodynamic model in which the transport of diffusing material can be simulated by the flow of a liquid through tubes. With this model the above problems can be solved by an alternative graphical method. Simple examples are given of the application of all three methods to experimental data.

L. P.

Hydrogen-bond-forming Powers of Atoms or Atomic Groups.

M. Tsuboi. *Bull. Chem. Soc. Japan*, **25**, 60-66 (Feb. 1952).
The degree of formation and strength of hydrogen bonds between proton donors X-H and proton acceptors Y in carbon tetrachloride soln. are measured from infrared absorption curves, where X-H is phenol, methanol, guaiacol, o-nitrophenol, methyl salicylate, salicylaldehyde, or methyl p-hydroxybenzoate; and Y is ethyl ether, hexamethylenetetramine, benzaldehyde, anisole, nitrobenzene, or methyl benzoate. It is not possible, generally, to calculate the energy of formation of intermolecular hydrogen bonds, and the frequency of the bonded H...Y band is used as a measure of the bond strength. In general, the strength of the hydrogen bond increases as the electron density on the hydrogen of X-H decreases (i.e. with increasingly electronegative substituents in X) and that on Y increases (i.e. increasing electronegativity of the group Y).

A. J.

Evidence of Intramolecular Attraction between Hydroxyl and Carbonyl Oxygen Atoms.

G. A. Jeffrey and G. S. Parry. *Nature*, **169**, 1105-1106 (28 June 1952).

Equilibrium Absorption of Direct Dyes by Cellulose Materials.

H. A. Standing. *Chem. and Ind.*, 527-530 (14 June 1952).
A review. The theories of Hanson, Neale, and Stringfellow (*Trans. Faraday Soc.*, **31**, 1718 (1935)), Willis, Warwicker, Standing, and Urquhart (*ibid.*, **41**, 506 (1945)), Crank (*J.S.D.C.*, **61**, 293 (1947)), and Peters and Vickerstaff (*Proc. Roy. Soc.*, **192A**, 292 (1948)) are compared.

A. J.

Desorption of a Direct Cotton Dye from Cellulosic Fibres.

J. H. E. Jackson and H. A. Turner. *J.S.D.C.*, **68**, 345-352 (Sept. 1952).

Donnan Membrane Equilibrium and Direct Dyeing of Cellophane—1 and II.

K. Nishida. *J. Soc. Textile Cellulose Ind. (Japan)*, **7**, 539-544 (1951); *Chem. Abs.*, **46**, 6389 (10 July 1952).

The amounts of Diamine Blue 3B absorbed by Cellophane from a bath at 90°C. containing 0.5 g. dye and 1-35 g. NaCl per litre agreed with the values calculated from the Neale equation derived from Donnan equilibrium, provided that the water adsorbed on the Cellophane, which acted as the solvent, was assumed to be 22/100 g. Cellophane, the amount actually adsorbed on Neale's cotton. Similar absorption was observed with Diamine Blue 2B, but here the actual amount of water adsorbed on the Cellophane, i.e. 93/100 g., had to be used to obtain agreement.

C. O. C.

Theory of Dyeing Cellulose Fibres by Direct Dyes.

1—Effect of Functional Groups on the Dye Uptake by Cellulose. F. I. Sadow and K. G. Kalina. *Kolloid Zhur.*, **14**, 118-123 (1952); *Chem. Abs.*, **46**, 6388 (10 July 1952).

Bleached cotton, containing 0.2% (of theoretical amount) CHO and 0.1% COOH groups, took up 0.21 g. Direct Pure Blue per 100 g. from a solution of 0.03 g. dye and 0.3 g. NaCl per litre at 55°C.; after 48 hr. the absorbed amount x increased to 0.24, and from a solution of 100 g. NaCl per litre x was 0.58 after 1 hr. The dialdehyde derivative of cellulose prepared from the bleached cotton and HIO₄ took up little more dye than did the original cotton as long as the COOH content was < 0.15% (the CHO content being 0.6-3.3%); when the COOH content was 0.5%, and the CHO content 12%, x was 10% of that of bleached cotton after 1 hr. and 50% after 48 hr. This decrease of x seems to be due to the low pH of the bath when the COOH content is great and to formation of lactol bonds. Monocarboxyl cellulose prepared from the bleached cotton and NO took up no dye from a bath containing 0.3 g. NaCl per litre, and its x from 100 g. NaCl per litre was smaller than x of the bleached cotton, and smaller the greater the COOH content; e.g. $x = 0.18$ after 1 hr. when the fibre contained 1 COOH per 2 glucose radicals. Monocarboxyl cellulose treated for 16 hr. with 0.05 N. calcium acetate took the dye up well, and x was often greater the greater the COOH content. Treatment with Ca acetate increased the pH of the bath, and so presumably caused the better dyeing. Xylan took up no dye from a liquor of 0.03 g. dye and 0.3 g. NaCl per litre, which shows that the CHO group of cellulose is important for dyeing.

C. O. C.

Prevention of the Reduction of Direct Dyes by Dyeing and on Steaming by the use of Sustilan N.

H. Hansen. *Textil Praxis*, **7**, 536-538 (July 1952).
Many direct dyes on cotton and other cellulosic materials are reduced in dyeing or on subjection to treatments involving the use of steam in the presence of small amounts of alkalis. This defect can be overcome by the addition of Sustilan N (FBy), which is a mixture of ammonium salts and amides of higher fatty acids, sulphonic acids, and substituted carboxylic acids of anion-active character.

B. K.

Practical Application of Vat Dyes to Yarn Dyeing.

S. Ikeda and Y. Fukui. *J. Soc. Textile Cellulose Ind. (Japan)*, **7**, 21-23 (1951); *Chem. Abs.*, **46**, 6389 (10 July 1952).

Viscose and cuprammonium rayon yarns, dyed by the pigment method with Indanthren Blue RRN, absorbed the dye levelly at a nearly constant rate to saturation in 20-25 min. The method is not yet applicable to dyes having large particles or those which require a special process, e.g. Indanthren Brilliant Pink R.

C. O. C.

Attachment of Dyes to Unimolecular Layers of Protein.

C. Wunderly. *Experientia*, **7**, (8), 296-297 (1951); *J. Textile Inst.*, **43**, A 410 (June 1952).
An ordinary Langmuir trough for the spreading of proteins is divided into two equal sections by a glass barrier. Protein monolayers can easily be transferred, from the surface on which they are spread, to the adjacent surface, where combination with dyes (e.g. Evans Blue, Trypan Red, Trypan Blue, Congo Red, Naphthol Yellow) takes place. The coloured protein is compressed to a narrow strip across the trough, and removed from it by inserting a 1 cm. wide glass slide under the strip and carefully lifting it. Excess water is removed by filter paper, and

the coloured protein is dissolved in 0.1N-NaOH. The solution is then transferred to a Beckmann spectrophotometer, and the absorption maxima for the protein and for the dye are measured. The extent of combination of the protein and the dye would seem to be dependent on the degree of dispersion of the latter, and a colloid-chemical reaction between the protein monolayer and the azo dyes is indicated.

C. J. W. H.

New Method of pH Control in Dyeing and the Metachrome Process. R. J. Hannay, W. H. Major, and R. Pickin. *J.S.D.C.*, **65**, 373-380 (Oct. 1952).

Investigation on Fur Dressing and Finishing. V.—Lake Formation during Oxidation Dyeing. F. Stäthler and R. Schubert. *Ges. Abhandl. deutsch. Lederinst. Freiberg/Sa.*, (6), 50-65 (1951); *Chem. Abs.*, **46**, 5852 (25 June 1952).

A study of the effect of varying the conditions when dyeing mordanted fur with oxidation dyes. The mordant solution was poured on to a 15% gelatin containing 0.2% sumac, and depth of penetration of the metal was measured after 2 hr. After rinsing and treating with dye liquor containing H_2O_2 , the depth of colour penetration was again measured after 24 hr. Penetration of metal increased with concentration and was twice as great for $K_2Cr_2O_7$ as for $CuSO_4$, $FeSO_4$, or potash alum. Penetration of dye was greater when the gel was treated with *p*-phenylenediamine before $K_2Cr_2O_7$, $FeSO_4$, or potash alum, but with $CuSO_4$ the order of treatment had no effect. pH of mordant had no effect. Dye penetration decreased with increasing mordant concn. for $K_2Cr_2O_7$, but increased with the concn. of $CuSO_4$, $FeSO_4$, or potash alum. Dye penetration decreased with increasing concn. of H_2O_2 for $K_2Cr_2O_7$ and $CuSO_4$, increased with H_2O_2 concn. for potash alum, and passed through a maximum with increasing concn. of H_2O_2 for $FeSO_4$. Dye penetration increased with increasing concn. of *p*-phenylenediamine most markedly for gel mordanted with $K_2Cr_2O_7$. Comparison of *p*-phenylenediamine with four other oxidation dyes showed little difference in penetration, except that *p*-aminophenol showed much less penetration with $K_2Cr_2O_7$ and potash alum, and Ursol F showed very low penetration with $FeSO_4$. The amount of lake formed on mixing solutions of *p*-phenylenediamine and mordants was measured after 20 hr. For a constant amount of *p*-phenylenediamine the amount of precipitate and % metal in it increased with concn. of $K_2Cr_2O_7$ (0.25-2.5 g. CrO_3 per litre). With $CuSO_4$ the effect was similar but smaller. With $FeSO_4$ the amount of precipitate and its Fe_2O_3 content increased when the amount of $FeSO_4$ was increased from 0.25 to 0.5 g. Fe_2O_3 per litre and remained unchanged for higher concn. With potash alum the amount of precipitate and its Al_2O_3 content were at a maximum at 0.5-1.0 g. Al_2O_3 per litre. Keeping the amounts of *p*-phenylenediamine and mordant constant, the amount of precipitate always increased, but the proportion of metal in the precipitate decreased with increase in H_2O_2 concn. For constant concn. of mordant, the amount of precipitate increased with concn. of *p*-phenylenediamine, but the proportion of *p*-phenylenediamine precipitated decreased and so did the proportion of metal in the precipitate. In all the above cases the amount of precipitate decreased in the order $K_2Cr_2O_7 > CuSO_4 > FeSO_4 > potash alum$, but the proportion of metal in the precipitate decreased in the order $FeSO_4 > potash alum > K_2Cr_2O_7$ and $CuSO_4$. Degreased wool and rabbit hair killed with Na_2CO_3 and NH_4OH , mordanted for 24 hr., and then dyed for 24 hr. with *p*-phenylenediamine, H_2O_2 being added after 1 hr., gave more level dyeing than the reverse treatment. With wool the depth of the dyeing increased with the amount of mordant used, but the handle was impaired. Varying the pH had no effect. Intensity of the dyeing increased with concn. of *p*-phenylenediamine to a maximum at 2 g. litre, i.e. 2 g./10 g. hair. Increasing the concn. of H_2O_2 (30%) to 2 ml./litre had an unfavourable effect on wool and no effect on hair. The amount of metal in the fibre increased linearly with the amount of $K_2Cr_2O_7$ used, and increased, but to a much smaller degree, with increasing amounts of $CuSO_4$, $FeSO_4$, and potash alum. Increase in pH and increasing concn. of H_2O_2 resulted in less metal on the fibre. The tensile strength of hair and wool decreased slightly, and elongation at break decreased considerably with increasing amount of mordant and/or H_2O_2 .

C. O. C.

New Developments in Dyeing Cellulose Acetate

Yarn. D. G. Carmichael and W. R. Ivey. *Amer. Dyestuff Rep.*, **41**, P 424-P 425, P 428 (7 July 1952).

A survey of acetate rayon dyeing includes an account of applying naphthols and fast colour bases simultaneously on acetate yarn in circulating machines, after which the base is diazotized to permit coupling in a second step, to give good light and wash fastness.

J. W. B.

Dyeing of Cellulose Acetate. IV—Dyeing with Indigo. R. Tanaka and T. Shinohara. *J. Soc. Textile Cellulose Ind. (Japan)*, **7**, 510-512 (1951); *Chem. Abs.*, **46**, 6389 (10 July 1952).

Adding acetic acid in increasing amounts while raising the temperature of the vat results in acetate rayon being dyed darker than viscose rayon at 50°C, when not less than half the NaOH in the bath had been neutralized. This effect seems to be due to increase of relative solubility of the indigo in the acetate rayon.

C. O. C.

Coloration of Activated Clays by Organic Vapours. H. Bakstain and F. Wieden. *Kolloid-Z.*, **125**, 173-174 (March 1952).

A wide range of activated clays (decolorizing earths, bentonites, etc.) become reddish coloured after heating for 2-5 weeks at 105°C. in the vapour of xylene, toluene, or, to a slight extent, tetralin. The colour resists extraction with organic solvents, reduction with iodine vapour in the cold, or oxidation with 3% hydrogen peroxide. The product reacts with alkalis and mineral acids as a reversible acid-base indicator, changing from red to yellow. Benzene, cyclohexane, decalin, aniline, methanol, ethanol, and isomyl alcohol do not show this effect. The aromatic nucleus is not, therefore, involved, and evidence is assembled to suggest that the activated earth induces oxidation of the hydrocarbon side-chain.

L. P.

PATENTS

Pigment Colouring. Imperial Paper & Color Corpn.

USP 2,546,188

The surface to be coloured is treated with an aqueous suspension of a pigment and then with an aqueous dispersion of a water-dispersible resin carrying electrical, preferably positive, charges, in presence of a substance which flocculates the resin.

C. O. C.

Photo-dyeing of Textiles, etc. L. E. Ravich.

BP 674,642

The material is treated with an aqueous solution of an ester of a leuco vat dye in presence of a compound which improves its solubility, e.g. disodium succinate, and a thiocyanate as sensitiser. The material is then exposed to light in presence of moisture, so as to precipitate the vat dye in the exposed portions.

C. O. C.

Surface Films of Azo-proteins (XII p. 419).

IX—PRINTING

Mechanical Theory of Printing Thickeners. W. Reif. *Melliand Textilber.*, **33**, 532-535 (June 1952).

An attempt is made to describe the printing properties of a print paste in terms of exact physical concepts. Good furnishing properties are associated with the property of the paste of being drawn out into a long thread, and here the theory of the tensile properties and of the fracture of plastic bodies is required. Printing behaviour is determined also by the viscosity properties of the paste—there are the usual rheological effects and, in addition, an internal "volume" viscosity effect, which comes into operation as a resistance to change in volume when the paste is compressed within the engraving during the printing operation. The author invents the term "Klebrigkeit" ("adhesiveness") for this effect. In a mathematical appendix, various relevant equations in elasticity, viscosity, and plasticity theory are brought together and discussed.

A. E. S.

Starches and Metal Salts in Printing. R. Haller.

TIBA, **28**, (4), 11-15 (1951); *J. Textile Inst.*, **43**, A 417 (June 1952).

It is shown that iron, aluminium, and chromium salts form insoluble compounds with the cell wall of the starch grain and prevent access of malt diastase, so that the efficiency of the latter as a means of removing starch thickening agents after printing is doubtful when these metals are present in the form of pigments. The situation

is alleviated somewhat by the presence of gum tragacanth, which dilutes or disperses the starch and tends to minimise the extent of coating of the cell wall. It should, however, be remembered that gum tragacanth is very unreliable as regards homogeneity.

C. J. W. H.

Problems in Screen Printing with Vat Dyes. I—Drying. G. Bernardy. *Melliand Textilber.*, 33, 628-633 (July 1952).

Screen printing with vat dyes gives rise to problems originating from premature decomp. of sodium formaldehyde-sulphoxylate. In direct printing, these difficulties can be avoided, and other advantages can be gained by using the Coloursun process, but this method is not applicable to discharge styles. Wasting of sulphoxylate during drying of the print may be due e.g. to an excessively high temp. and/or humidity in the print-shop, to the presence of acid vapours, to the presence in the print paste of catalytic impurities, e.g. Cu and Fe compounds, or to a slow rate of drying. Means of drying are surveyed, including hot air, with and without pre-drying, radiant heat, and the use of hot tables. The use of predried hot air is particularly to be recommended for the present purpose. Other precautions recommended are the use of thickenings of high solid content and the addition of certain substances, e.g. formaldehyde and Zn dust, to the print paste.

A. E. S.

Dye Gelatin Phosphors. D. Yamamoto and R. Iwaki. *J. Chem. Soc. Japan, Pure Chem. Sect.*, 72, 1075-1078 (1951); *Chem. Abs.*, 46, 5974 (10 July 1952).

Films of gelatin containing acridine, diphenylmethane, triphenylmethane, xanthene, cyanine, azine, thiazine, and alloxan display phosphorescence with a lifetime of not > a few tenths of a second, some of them showing positive pre-exposure effect. Azo dyes are not phosphorescent.

C. O. C.

Studies on the Magnetic Dichroism of Dye-Gelatin Films. III—Magnetic Dichroism of Dye-Gelatin Films excited by Light. IV—Confirmation of Photomagnetism by means of Magnetic Dichroism in Dye-Gelatin Films excited by Light. D. Yamamoto. *Bull. Chem. Soc. Japan*, 24, 211-214; 214-218 (Nov. 1951).

III—The magnetic dichroism of dyed gelatin films is measured before and after illumination by a suitable light source. Considerable differences are found with Malachite Green and Rhodamine B, and to a lesser extent with some cyanine and azo dyes. The effects may be due to photomagnetism.

IV—A theoretical treatment of magnetic dichroism-time relationships for excited molecules assuming a photo-magnetic effect. The results agree qualitatively with the experimental results (Part III), but are not conclusive.

A. J.

PATENTS

Continuous Printing with Vat Dyes. DuP. *USP* 2,587,905

The dye is printed on in a thickened paste and dried. The entire fabric is then overprinted with aqueous caustic alkali and sodium hydrosulphite, and then within 20 sec. of this overprinting passed into an air-free steam chamber, where reaction and fixation of the dye occur. The material is then oxidised, soaped, etc. as usual. Fast prints with sharp outlines and unstained whites are obtained.

C. O. C.

Removal of Phthalocyanine and other Pigments from Printed Textiles. Arkana Co. *USP* 2,587,597

Phthalocyanine pigments embodied in water-insoluble resins can be removed by treatment in a bath containing sodium hydrosulphite, caustic soda, and an aliphatic quaternary compound preferably containing 1 alkyl chain of 14-22 C and 3 alkyl chains of 1-3 C each; if desired, anthraquinone may be included.

C. O. C.

Imbibition Printing of Cinematograph Film. Technicolor Motion Picture Corp. *BP* 676,952

Dyed matrices are pressed successively against a dye-absorptive blank in several stages, in each of which the blank and one matrix are continuously fed together and thence in contact with one another along a predetermined path, the blank being wetted before each dye transfer to make it absorb more quickly. Each wetting after the first

dye transfer is effected entirely by spraying liquid against the face of the blank and so avoiding submergence of the blank in liquid.

C. O. C.

Pigment Colouring (VIII p. 415).
Photo-dyeing of Textiles, etc. (VIII p. 415).

X—SIZING AND FINISHING

Formation of Cellulose Ethers in Water-repelling Treatment of Cotton with Stearamidomethylpyridinium Chloride. H. A. Schuyten, J. W. Weaver, J. G. Frick, and J. D. Reid. *Textile Research J.*, 22, 424-432 (June 1952).

Stearamidomethylpyridinium chloride is shown to react with cellulose hydroxyl groups to produce a stearamidomethyl ether of cellulose. The permanent water-repelling obtained on treatment of cotton fabrics with amidomethylpyridinium salts is shown to be due to this reaction.

A. B.

Stabilisation Problems in Textile Materials. R. Ferry. *Industrie text.*, 68, 587-590 (Dec. 1951); *J. Textile Inst.*, 43, A 420 (June 1952).

Treatment of fabric mixtures containing cellulose fibres with hydroxymethylformamide was found to give good protection against both shrinking and creasing. The formaldehyde is strongly bound in this compound and formaldehyde fumes are not given off, as was the case with bis-hydroxymethylformamide.

C. J. W. H.

Correlation of Affinity for Wool of Water-soluble Substances with their Bactericidal Efficiency. S. Seidenberg, M. Erne, and R. Fischer. *Experientia*, 6, (5), 192-193 (1950); *J. Textile Inst.*, 43, A 422 (June 1952).

It has been found that the higher the affinity of a water-soluble substance for wool at pH 7, the higher is its bactericidal activity. This correlation has now been demonstrated for various chemically differing substances. The correlation exists separately within the anion-active group and within the cation-active group of substances. Addition of salt increases the affinity of a substance for wool, and simultaneously the bactericidal activity of the substance is increased. The determination of the affinity of a substance for wool permits the prediction of the approximate bactericidal (not the bacteriostatic) activity of this substance. It is assumed that the existence of the correlation is due to the cystine content of wool.

C. J. W. H.

Heat-setting of Synthetic Fibres. F. Fourné. *Melliand Textilber.*, 33, 639-643 (July); 753-757 (Aug. 1952).

Much general information concerning the production and properties of the more important synthetic fibres is given in the form of tables. The theory of setting, including the effect on setting of the presence of swelling agents, is given, and the optimum conditions for the setting of Perlon L (P), nylon (N), Orlon (O), and Terylene (T) are stated. The optimum setting temperatures (°C.) when using dry heat (which includes heating by means of non-aqueous liquids or superheated steam) are P—190, N—225, O—~200, T—234; when the setting is done on the swollen fibre (saturated steam), they are P—130, N—131, O—134, T—126. For the success of the process, it is essential that after a short time at the setting temp. the fibre should be cooled rapidly, while in the desired geometrical form, to below a certain quenching temp. The quenching temperatures (°C.) are P—65, N—82, O—90, and T—85. The importance of ensuring even treatment is stressed; e.g. in the steam treatment of yarn packages, it is advisable to evacuate the steamer before introducing the steam, in order to aid penetration. When the synthetic fibres are mixed with e.g. viscose rayon, the potential shrinkage in the latter must be taken into account when considering the setting conditions. Machines used for setting yarn, cords, fabrics, and stockings are described and shown in illustrations, and some indication is given of the relative advantages in particular cases of setting by dry heat and by saturated steam. The resistance of various types of dye to the setting processes is discussed.

A. E. S.

Setting and Finishing of Nylon Textiles. G. K. Mecklenburgh, S. Shaw, and H. W. Peters. *J.S.D.C.*, 68, 381-391 (Oct. 1952).

PATENTS

Twist-setting of Nylon Yarn. British Nylon Spinners. BP 677,341

Continuously running nylon yarn is heat-set by passing it between the plates of a condenser to submit it to the action of a high-frequency electric field. C. O. C.

Preparing Bagged Fabric for Finishing in Open Width. Celanese Corp. of America. BP 674,782

The bagged fabric is continuously hydroextracted in flattened form, opened out to full width after withdrawing the stitches, and again hydroextracted. Hydro-extraction is preferably effected by suction. The three processes are operated successively as a continuous whole. C. O. C.

Relaxation Shrinkage of Flat-knit Fabric. R. Evans. USP 2,588,624

The material is bagged and then, while still wet from scouring or dyeing (or if necessary it is wetted out), passed into a machine in which the courses and lateral edges are straightened and all internal stresses relaxed, and is finally dried while held between two layers of other fabric, e.g. two endless conveyors passing around a drying cylinder. C. O. C.

Imparting Dimensional Stability to Woven Cotton Fabrics. American Cyanamid Co. BP 674,645

Dimensional stability is imparted to cotton cloth without decrease in tensile strength by impregnation with a colloidal aqueous solution of a polymerized, positively charged condensate of an aldehyde and an aminotriazine having $> 1 \text{ NH}_2$ group and an H atom attached to each amino N atom. The solution must contain 0.5–7.0 mol. of a water-soluble, saturated aliphatic mono- or hydroxy-mono-carboxylic acid (of dissociation constant 1.4×10^{-4} – 2.5×10^{-4}) per mol. of monomeric aminotriazine-aldehyde condensate. The fabric should pick up 2.5–15.6% of its dry weight of the condensate. On drying, the condensate is deposited in the fibre in a water-insoluble form. C. O. C.

Disrupting the Cross-linkages in Keratinous Fibres. N. Chambers. BP 674,586

Aqueous magnesium hydrosulphide disrupts the cross-linkages of keratin, the effect being appreciable at concentrations equivalent to 0.25% MgO, and greater concentrations causing greater disruption. The effect is increased if enough MgCl_2 is present to form compounds of the type $\text{MgCl}_2 \cdot 1-6 \text{ MgO}$. C. O. C.

Chlorinating Wool. Ciba. BP 675,137

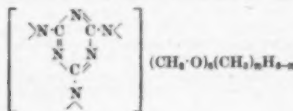
In the aqueous chlorination of wool, more even treatment is obtained if a water-soluble hydroxymethyl-melamine or a water-soluble alkylated, especially methylated, hydroxymethyl-melamine is present. C. O. C.

Wash-fast Stiff Finish. Monsanto. USP 2,586,477

The material is treated with an aqueous solution or dispersion of an alkali or amine salt of copolymerized styrene-maleic anhydride and then with a heavy metal salt. The "heavy" metal salts mentioned in the claims are those of Zr, Al, and Cu. C. O. C.

Glazed Finish. Monsanto. BP 674,948

A soft, durable, and wash-resistant glazed finish is obtained by treating fabric with an aqueous solution or dispersion of a resin precondensate of formula—



($n = 5$ or 6), drying the fabric until it retains 9–20% moisture on its dry weight, glazing mechanically, and then heat-curing in presence of a catalyst. C. O. C.

Water-repellent Finish. Dow Corning Corp.

Fibres are rendered water-repellent by impregnating them with a mixture of (a) 20–70% by wt. of a liquid methylpolysiloxane, containing 2.0–2.1 CH_3 per Si atom and of viscosity 1000–100,000 centistokes and (b) 80–30%

of a liquid methylhydrogenpolysiloxane containing 1.0–1.5 CH_3 radicals and 0.75–1.25 H atoms bonded to Si per Si atom, there being 2.0–2.25 ($\text{CH}_3 + \text{H}$) per Si atom, and then heating for 3 sec.–1 hr. at 100–475°. The finish is applicable to fibres of all types, gives a soft handle, and does not tend to show mark-off.

USP 2,588,366

A mixture containing 10–30% of the methylpolysiloxane, 12–45% of the methylhydrogenpolysiloxane, and 20–65% of a resin of formula $(\text{CH}_2)_n\text{SiO}_{2-n}$ ($n = 1.0-1.25$) is used. An excellent water-repellent finish is imparted without the fabric acquiring a slippery handle. C. O. C.

Rendering Cellulosic Material Non-adherent to Adhesives. Dow Corning Corp. USP 2,588,367

Cellulosic material is rendered non-adherent to adhesives by treating it with a liquid of formula $(\text{CH}_2)_n\text{H}_2\text{SiO}_{2-n}$ ($n = 1.0-1.5$; $b = 0.75-1.25$; $a + b = 2.0-2.25$) dispersed in an aqueous solution of a cellulose ether. C. O. C.

Deep Embossed Effects on Fabrics. C. A. Redfarn. BP 675,207

Embossed effects resembling quilting are obtained by impregnating with a thermosetting resin or its precondensate, drying, and then passing between two embossing rolls, one of which has a reverse contour to the other so that they co-operate with close-mating intermesh. The embossed cloth is then heat-cured. C. O. C.

Bonding Cellulose to Rubber. Courtaulds. BP 676,703

Better bonding between cellulose and rubber is obtained by use of a phenol-aldehyde condensate having aliphatic side-chains containing an ethylenically unsaturated carbon-carbon linkage, as the adhesive prior to vulcanisation. C. O. C.

Coating Fabrics with Cellulose. Textuff. BP 675,932

The fabric is steamed to eliminate the air absorbed in it, then passed through a bath of a regeneratable cellulose derivative, and finally treated with a coagulant. The cellulose-derivative liquor is continuously withdrawn from the treating bath, filtered, and returned to the bath. C. O. C.

Laminated Fabric. Morton Sundour Fabrics. BP 674,853

Fabric which remains flexible at -80 to 150°F , is resistant to mineral oils and chemicals, is proof against micro-organisms and all pests, and is as strong as aluminium of equal thickness consists of at least two layers of nylon cloth one of which has lower m.p. than the other or of nylon cloth and a nylon material, e.g. moulding powder, of lower m.p., the whole being bonded together by pressure and heated so as to melt the layer of lower m.p. and yield a product which is impervious to liquids. C. O. C.

Stiffened Bonded Fabrics. L. Kanitz. BP 673,922

When bonding fabrics by means of thermoplastic resins, a much thinner film of the adhesive is needed, or alternatively a much more loosely woven interlining which does not take on so much adhesive can be used, if before heating and pressing the assembly is treated with a dilute aqueous solution of one or more of the following—diacetone alcohol, diethylene glycol, ethyl, butyl, or methyl lactate, acetonylacetone, ethylene glycol monoethyl ether acetate, diethylene glycol monoethyl ether, ethylene glycol monomethyl ether, dipropylene glycol, diethylene glycol monoethyl ether acetate, and diethylene glycol monoethyl ether. Preferably the solution also contains as plasticizer mono-, di-, or tri-acetin, diethylene glycol diacetate, dimethoxytetraglycol, triethyl phosphate, and diethyl tartrate. The solution contains 10–20% of solvent and plasticizer. The bonded fabric is resistant to laundering at the boil.

BP 674,024

Addition to the thermoplastic resin of not < 15% of a urea-, thiourea-, or melamine-formaldehyde condensate by adding the precondensate or one of its components to the damping liquid gives increased resistance to laundering. A small amount of a gelatin-formaldehyde compound considerably increases the effect.

BP 674,831

Addition to the damping liquid of 0.5–3% of a thermoplastic substance which increases the resistance of the

fabric to abrasion, e.g. polyvinyl acetate or polymethyl methacrylate, results in a product having better resistance to wear. C. O. C.

Pile Fabric from Adhesive Chenille Threads. G. C. Alier, A. V. Champagnat, and J. F. M. Tirmont.

BP 674,922

Textile threads are coated with rubber latex containing vulcanisation agents or with suitable synthetic resins or adhesives, and are superimposed in parallel form across a sheet of parallel non-coated threads, and pressure is applied to produce adhesion. These non-coated threads are then cut in lines parallel to and between the coated ones, and the sheets passed over a heated channelled cylinder to produce the usual U-shaped chenille. Heating is insufficient completely to harden the adhesive, which is required for the next stage, in which parallel chenille threads, made as above or conventionally, are further coated underneath with suitable adhesive and passed on to a backing sheet of e.g. unvulcanised rubber, coated felt, or adhesive composite material. After firm pressing, the assembled carpet is passed into a tunnel-stove for vulcanisation or hardening. J. W. B.

Gaseous Chlorination of Wool Garments. Forstmann Woolen Co. USP 2,590,811

Wool garments containing 10-5-12-0% moisture can be chlorinated evenly and without hydrochloric acid damage by suspending each garment individually from a hanger having a non-condensing, hygroscopic surface and low heat capacity. Wool-covered hangers are best. Greater reduction of felting property is obtained than if chlorination is carried out at < 10% moisture content, and there is less loss in strength. C. O. C.

Deposition of Polymers on the Wool Fibre and Allwörden's Reaction (VI p. 410).

Polyisocyanates in Bonding. I—Terylene to Rubber (XIII p. 419).

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Colloid-chemical Properties of Water-soluble Methyl Celluloses. J. Stawitz, *Mollard Textilber.*, 33, 620-623 (July); 737-738 (Aug. 1952).

A brief discussion of the physical properties of aq. methyl cellulose solutions is followed by a very full factual and theoretical exposition, based on the available literature, of their rheological behaviour, as influenced by the shearing force, the concn., the temp., and the addition of electrolytes. There are 50 references. A. E. S.

Behaviour of Membranes between Electrolyte Solutions. III—Dissociation of Carboxyl Groups in Cellulose. P. Hirsch, *Rec. Trav. chim.*, 71, 525-544 (May 1952).

Samples of Cellophane and various oxycelluloses from filter paper are titrated against NaOH, and back-titrated against HCl. The initial neutralisation curve with NaOH differs from the back-titration curve. Subsequently, both curves are identical and reproducible. A prolonged treatment with NaOH before titration also leads to reproducible curves. The shape of the curves varies with the degree of oxidation, approaching the theoretical curve for a monobasic acid with more highly oxidised cellulose. The presence of electrolyte also affects the shape of the curves. The initial difference between forward and back titration curves is thought to be due to formation of lactones, which are hydrolysed by alkaline treatment. The law of mass action and the Donnan equilibrium are not sufficient to explain the shape and position of the neutralisation curves, and there is no clear connection with the membrane diffusion potential. A. J.

Sodium Salts of Pectins and of Carboxymethyl Cellulose in Aqueous Sodium Chloride. III—Potentiometric Titration. D. T. F. Pals and J. J. Hermans, *Rec. Trav. chim.*, 71, 513-520 (May 1952). Solutions of sodium pectinate and carboxymethyl cellulose containing varying amounts of NaOH or HCl and

sometimes NaCl are titrated potentiometrically against HCl or NaOH, and resulting values of pK are plotted against degree of ionisation α of the polymer. The experimental values of $\Delta pK (= pK - pK_0)$ obtained are about 100 times greater than the predicted values. Possible causes of this discrepancy are—(a) necessity to distinguish between $[H^+]$ inside and outside the polymer coils; (b) fluctuations in the end-to-end distance of polymer molecules are ignored; and (c) uncertainty in the activity coefficients used, which will be affected by the presence of polyelectrolytes. It is considered that potentiometric titrations are of little value in the study of molecular dimensions unless the charge density in the polymer is high. A. J.

PATENTS

Coating of Paper. Mead Corpn.

BP 676,867

A smooth and finished coating is produced by applying a fluent coating material to paper, and while the coating is still mobile and fluent treating it with a smoothing and finishing member. The adhesive characteristics of the coating are modified, without removal of fluid from it, while in contact with the smoothing member, so as to complete release and separation of the coated paper from the surface member while retaining the smooth finish of the coating. C. O. C.

Applying a Discontinuous Coating to Paper. North American Paper Process Co. USP 2,589,302

Paper which appears to be uniformly coated is produced by directly printing on to it closely spaced but segregated and uniform quantities of an aqueous mineral pigment coating mix including a binder, water, and an inorganic filler. The binder—starch or casein—is 7-1-9-2% by wt. of the mix and the filler 45-7-47-6%. While the coating is still moist, it is treated by a highly polished rotating glazing roller travelling at the same speed as the paper with pressure such as just ensures uniform even contact. The segregated portions of mix maintain their separateness but are partly flattened. The glazing roller is heated to such a temperature that part of the binder is caused to migrate on the uncoated paper and is then glazed by the roller to form a continuous skin over the paper. C. O. C.

Glossing Paper. United States Rubber Co. BP 675,452

High gloss is continuously imparted to paper by feeding paper, coated on one side with an aqueous dispersion of a thermoplastic resinous polymer having a second-order transition temperature (T_g) of 55-125°C. and subsequently dried and baked at 20-60°C. above T_g to set the polymer on the surface, into the nip of a continuously rotating, hard, non-resilient, mirror-finished glossing roll and a co-operating continuously rotating, resiliently surfaced roll, rotating at a speed identical with it, with the coated side of the paper in contact with the mirror-finished roll, passing the paper web round the latter in intimate contact for a substantial angular distance ($< 180^\circ$), heating the coating to $(T_g + 60 \pm 20 - 0.4 P)^\circ C.$ ($P = \%$ of plasticiser by weight) while in contact with the mirror-finished roll, so that the heat and pressure (200-1500 lb./sq. in.) exerted cause the coating to flow into a thin, highly glossed, tenaciously adherent, continuous film, continuously stripping the hot paper from the hot mirror-finished roll by withdrawing it tangentially, and continuously passing it along a straight path until the coating cools to such an extent that its gloss is not injured by bending. The thermoplastic polymer is a styrene-acrylonitrile copolymer (65:35 to 80:20 by wt.). The aqueous dispersion contains 50-95% of the thermoplastic polymer and 5-50% of a butadiene-acrylonitrile copolymer. The amount of the polymer applied to the surface of the paper web ranges from 0.5 to 4.0 lb./1000 sq. ft. S. V. S.

Decorative Laminæ. H. J. Mallabar. BP 674,149

Modification of BP 653,531 (*J.S.D.C.*, 67, 325 (Aug. 1951)). Paper is coated with a white or coloured pigmented filler, the coating brushed to ensure uniform distribution, and the coated paper impregnated with a thermosetting resin. The pigmented filler may be partly or wholly replaced by a light-reflecting particulate material, which may be a metallic powder, e.g. bronze or aluminium, or a fish-scale product, e.g. H scale (pearl essence). S. V. S.

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Physical and Physicochemical Constants of Leather and Collagen. C. E. Weir. *J. Soc. Leather Trades Chem.*, **36**, 155-163 (May 1952).

Function of the Carboxyl Groups of Collagen in the Fixation of Cationic Chromium Complexes as shown by the Behaviour of Carboxylic-type Cation Exchangers towards Chromium Compounds. K. H. Gustavson. *J. Soc. Leather Trades Chem.*, **36**, 182-201 (May 1952).

The fixation of cationic chromium of basic chlorides and sulphates by a carboxylic cation-exchange resin (Amberlite IRC-50) and by collagen is investigated. The resin fixes more cationic chromium than collagen, owing to displacement of complex acid groups (e.g. sulphate) from the chromium complex by the resin. There is a striking similarity between the resin and collagen in reactions with complexes in which the cationic chromium is the dominating constituent, supporting the concept that normal chrome tanning principally involves carboxyl groups in collagen and electropositive Cr. With complexes containing anionic and non-ionic Cr (e.g. extremely basic chlorides) the resin fixes much less Cr than does collagen, owing to non-ionic fixation by the protein. This is further illustrated by the use of aged solutions. The view that chromium complexes are always fixed by the carboxyl groups of collagen cannot be reconciled with the fixation of non-ionic phthalatosulphatochromium complexes and anionic tetraxalatochromate and hexathiochromate-chromate. A. J.

Violet Coloration on Flesh Side of Undyed Chrome Suede on Shaving. G. Otto. *Leder*, **3**, 45 (1952); *J. Soc. Leather Trades Chem.*, **36**, 202 (June 1952).

The coloured zones which are revealed on shaving are due to the presence of soluble protein bodies formed in liming, which have not been able to escape. These react with the chrome liquors, and are most noticeable on dyeing. They may be prevented by an aftertanning with basic aluminium chloride (Blancetan B). C. J. W. H.

Unshairing by means of Enzymes. G. H. Green. *J. Soc. Leather Trades Chem.*, **36**, 127-134 (April 1952).

This is a review of the literature divided into sections according to whether the enzymes used have been derived from animal, mould, bacterial, or higher plant sources, and into methods independent of the source of the enzyme. Sixty-one references are given. A. S. F.

Surface Films of Azo-proteins. T. Tachibana, K. Fukuda, K. Inokuchi, S. Yamoka, and K. Suzuki. *Bull. Chem. Soc. Japan*, **25**, 71-76 (Feb. 1952).

Azo-ovalbumin containing various numbers of azo groups is prepared by the action of diazotised *p*-phenylarsonic acid on purified hen ovalbumin. Monolayers are spread on 0.01 N-HCl or half-saturated ammonium sulphate soln., and force-area curves obtained by Wilhelmy's method. The limiting specific area decreases with increasing azo content, but plotting area per molecule instead of specific area is claimed to give identical curves for all azo-ovalbumins. The limiting area of one azo-ovalbumin molecule is given as 7000 sq. Å. It is considered that introduction of azo groups increases the thickness of the azo-ovalbumin monolayer but does not affect the area. The antigenic properties of built-up films of azo-ovalbumins are also investigated and discussed. A. J.

Investigations on Fur Dressing and Finishing. VI—Effect on the Hair and Leather of Bleaching Natural-coloured Furs (VII p. 413).

Investigations on Fur Dressing and Finishing. V—Lake Formation during Oxidation Dyeing (VIII p. 415).

XIII—RUBBER; RESINS; PLASTICS

Polyisocyanates in Bonding. I—Terylene to Rubber. T. J. Meyrick and J. T. Watts. *India-Rubber J.*, **122**, 467-468 (1952).

Casein and resorcinol-formaldehyde adhesives gave poor bonding, but satisfactory results were obtained with polyisocyanate adhesives. Better adhesion was obtained by spreading the rubber on the Terylene than by dipping. C. O. C.

Ultraviolet Absorption Spectra of High Polymers. G. Scheide and R. Fausa. *Kolloid-Z.*, **125**, 139-149 (March 1952).

A review of the structural interpretations deducible from absorption spectra in the ultraviolet region of long-chain compounds such as polystyrene, proteins, and polypeptides. L. P.

Vinyl Plasticiser Developments. R. G. Kadosch. *Modern Plastics*, **29**, (12), 111-118, 178-182 (Aug. 1952).

Review of recent developments in the use of plasticisers in vinyl plastics; 107 references. C. O. C.

PATENTS

Moistureproof Films. British Collophane. *BP* 669,907. Emulsions of polymers and copolymers of vinylidene chloride are converted to foils of low moisture transmission by casting on to a sheet of non-fibrous water-absorbent base, e.g. of regenerated cellulose, of such thickness as to absorb all the water present in the emulsion. The deposited film is removed and consolidated by heat. E. C.

Varnish Resins. ICI. *BP* 669,763. Resins which are reactive to drying oils are prepared by heating at 100-140°C. an alcohol-modified melamine-formaldehyde reaction product with a polymer of coumarone and/or indene. E. C.

Rapid-drying Varnish Resins. Pinchin, Johnson & Associates. *BP* 665,473.

Rapid-drying varnish resins are obtained by heating at 200°C. an alkylated hydroxymethylmelamine with a hydroxyester prepared from drying or semi-drying oil fatty acids and a polyhydric alcohol, e.g. glycerol or pentaerythritol. E. C.

Styrene-modified Drying Oils. Dow Chemical Co. *BP* 664,432.

Resins suitable for use in paints and varnishes are obtained by heating together at 100-300°C. a partly oxidised drying oil in which < one-quarter of the unsaturation is conjugated and a mixture of an alkenylbenzene (< 12 C), e.g. styrene, with less than an α -alkylalkenylbenzene (< 13 C), e.g. α -methylstyrene. E. C.

Ultraviolet Absorption of Silk Fibroin—1 (VI p. 410). **Plasticising Cellulose Acetate** (VI p. 411).

XIV—ANALYSIS; TESTING; APPARATUS

High-alkaline Colorimetric pH Determination. R. H. M. Simon. *Anal. Chem.*, **24**, 1215-1216 (July 1952).

An indicator dye is used whose visible light transmittance changes with pH sufficiently to allow the calibration in pH units of a suitable photocolormeter. The most satisfactory results have been obtained using Parazo Orange, and Tropaeolin O together with a light filter absorbing below 400 m μ . By this means pH values in the region of 12.5 may be determined, the transmittance of these dyes decreasing with rising pH. The dye concentration, which is critical, is kept exactly the same in both the soln. used for calibration and the unknown. J. W. D.

Microchemical Determination of Magnesium, Calcium, and Zinc with Disodium Ethylenediaminetetra-acetic Acid. E. W. Debnay. *Nature*, **169**, 1104-1105 (28 June 1952).

A modification of previously published methods. J. W. B.

Ester-fractionation Method for the Component Fatty Acid Analysis of Mixtures containing Hydroxy Fatty Acids. K. T. Achaya and S. A. Saksena. *Analyst*, **77**, 375-380 (July 1952).

An ester-fractionation method, not involving acetyl values, for the analysis of natural or synthetic mixtures containing hydroxy fatty acids is illustrated by reference to a synthetic mixture containing 70% by weight of the mixed fatty acids of groundnut oil and 30% of ricinoleic acid.

Saturated acids are removed by the usual lead salt-alcohol separation. Oleic-linoleic acid mixtures are not separated from ricinoleic acid, presumably because of the insolubility of the lead salts of ricinoleic acid in light petroleum owing to mixed salt formation. On the other

hand, a concentrate of oleic (containing linoleic) acid can be separated from one of ricinoleic (containing linoleic) acid by two precipitations of the urea-insoluble adducts of the oleic-linoleic mixture in methanol.

The composition of the original mixture is calculated from the iodine and saponification values of the fractionated methyl esters of the saturated and oleic-linoleic extracts, and of the fractionated acetylated methyl esters of the ricinoleic acid extract. Results agree well with theory. All experimental details are furnished.

J. W. D.

Analytical Chemistry of cyclohexanone. A. Castaglioli. *Z. anal. Chem.*, **135**, 110-113 (1952); *Chem. Abs.*, **46**, 5488 (25 June 1952).

Cyclohexanone in presence of NaOH gives deep blue with *o*-nitrobenzene if enough cyclohexanone is present. Cyclohexanone can be determined in presence of cyclohexanol by condensing with furfuraldehyde in presence of NaOH.

C. O. C.

Analytical Methods in the Starch and Dextrose Industry. W. H. Fetzner. *Anal. Chem.*, **24**, 1129-1137 (July 1952).

A review of analytical methods used in the modern starch and dextrose industry; these include—determination of solids content of starch slurries by density, and by refractive index; measurement of colour in corn syrup; determination of reducing sugars (including the determination of dextrose in the presence of other reducing sugars); estimation of moisture in starch and corn syrup; measurement of viscosity by various methods (including the recording of changes in rheological properties of starch pastes through a cooking and cooling cycle). There are 60 references, and 7 photographs of testing equipment.

J. W. D.

Dichroism of Dye Molecules in Polyvinyl Alcohol. S. Miyakawa, K. Hasegawa, and T. Uemura. *Bull. Chem. Soc. Japan*, **24**, 289 (Dec. 1951).

Correction to previous paper, *ibid.*, **23**, 260 (Dec. 1950); *J.S.D.C.*, **67**, 485 (Nov. 1951).

Differentiating Colour Test for Fluorene Derivatives. E. Sawicki. *Anal. Chem.*, **24**, 1204-1205 (July 1952).

Unlike its analogues, fluorene reacts on warming with acetone and KOH, to produce a blue-green colour. This colour results also when fluorene derivatives with an electron-attracting group in the 2-position are used. Similarly located electron-repelling substituents lead to a variety of less vivid colours (or ppt.) ranging from white to red-brown. Derivatives of fluorenone which contain *m*-electron attracting groups yield violet or purple colours with acetone in the cold, this reaction being shared by a number of other compounds, including *m*-dinitrobenzene. A different series of colours obtains when ethanol is used in place of acetone, which assists (together with the colours obtained with acetone) in distinguishing the fluorenone derivatives from the others sharing the acetone reaction. Tables are given in which the colours resulting from the above reactions are listed for a number of compounds. 2-Nitrofluorenone reacts with ketones containing an active methylene group (a number of these are listed also) to give a violet coloration, the reaction being sensitive enough to detect the ketone at a dilution of 1 in 10,000. J. W. D.

Measurement of Damage in Coarse Keratinous Materials. F. Kidd and C. S. Whewell. *J.S.D.C.*, **68**, 396 (Oct. 1952).

Identification of Man-made Fibres. P.-A. Koch. *Textil-Rund.*, **7**, 273-277 (June 1952).

Tabular synopsis of methods for the identification of cellulose rayons, alginate rayons, fibres from polyvinyl chloride, polyesters, polyamides, and polyacrylonitrile, and regenerated protein fibres. Identification is based in the main on microscopic examination of cross-sections of the fibres and their solubility in inorganic and organic solvents.

B. K.

Spectral Sensitivity of Light-fastness Standards. A. Luszczak and H. Zukriegel. *Melliand Textilber.*, **33**, 535-537 (June 1952).

German light fastness standards (No. 1, 3, 4, and 6) are exposed in a Heliotest (see *J.S.D.C.*, **68**, 235 (June 1952)) fitted with quartz lenses. Exposures are made to unfiltered daylight and to a number of wavebands covering the spectrum, selected by means of filters. The relation

already proposed (see *J.S.D.C.*, **68**, 268 (July 1952)) between wavelength λ of the ultraviolet absorption max. and the light-fastness grade is confirmed. It is noted also that, for each dye, fading by sunlight is to be attributed mainly to a particular band containing wavelengths appreciably $> \lambda$ and that this band has a fairly definite boundary at 2λ (the experimental values for No. 1, 3, 4, and 6 are 1.99 λ , 2.02 λ , 2.02 λ , and 1.96 λ respectively) separating it from longer wavelengths that are practically devoid of fading action. Thus, standard No. 1, a dyeing on wool of Brilliant Wool Blue FFR extra, which has an ultraviolet absorption max. at 3168 μ , within the limits of the spectrum of sunlight, fades appreciably when a filter with a range of 3084-3926 μ is used, does not fade at all with a filter of range 3400-3900 μ , fades almost as rapidly as in unfiltered light with a filter of 4100 μ max. transmission, and is not affected by light of wavelength $> 6300 \mu$ ($\sim 2 \times 3168 \mu$). Such variations in the spectral sensitivity of dyes make the well known variability in the results of light-fastness tests done under various conditions of natural illumination readily explicable. It is demonstrated that extreme conditions can be found (exposure to an electric welding arc through a filter that removes wavelengths $< 3160 \mu$) under which all the eight standards fade at practically the same rate. Exposure of one of the standards for a certain period of time to radiation obtained from a filtered ultraviolet source and having the range 3400-4000 μ , which is without appreciable fading action, leads to a definite darkening in colour. The original depth is restored on standing or, more rapidly, by further irradiation with the lamp or with unfiltered sunlight. Pre-irradiation in this way has no effect on the time required for just discernible fading to occur in sunlight. It is considered that the darkening produced by radiation of wavelength 3400-4000 μ is evidence of the formation of a labile intermediate substance. Such substances are considered to be formed also by radiation that produces fading, but they undergo further changes, in the course of which energy that was originally in the form of a definite number of small quanta is made available again, probably in the form of ultraviolet luminescence, as a smaller number of larger quanta. These quanta correspond to wavelengths within the ultraviolet absorption band of the dye and produce fading. This mechanism explains the existence of a relation between the position of the fading wave-band and that of its absorption band, and it explains fading action at a distance, which has been observed by various authors and is evidenced in some of the present experiments by the formation of a halo of partial fading at the edge of the masked portion of the pattern. It is considered improbable that the effect can be attributed to the formation of hydrogen peroxide, as suggested by Egerton (see *J.S.D.C.*, **65**, 324 (June 1949)).

A. E. S.

Quantitative Relationship between Depth of Dyeing and Light Fastness. J. C. Eaton, C. H. Giles, and M. Gordon. *J.S.D.C.*, **68**, 394-396 (Oct. 1952).

Photochemistry in the Lower Atmosphere. F. E. Blacet. *J. Ind. Eng. Chem.*, **44**, 1339-1342 (June 1952).

A brief description is given of the spectrum of the sun in the lower atmosphere. The photochemical reactions which occur are initiated as a result of light absorption by pollutants. The most important is the conversion of O_3 to O_2 through the medium of NO_x , a cyclic process in which a small amount of NO_x may produce much O_3 . The ozone so produced may react with other contaminants, e.g. unsaturated hydrocarbons and oxygenated organic compounds resulting from incomplete combustion of fuels, etc.

W. K. R.

Fastness to Perspiration. Society of Dyers and Colourists, Perspiration Fastness Subcommittee. *J.S.D.C.*, **68**, 392-394 (Oct. 1952).

Modified Perspirometer. S. V. Vaeck. *Textile*, **17**, 465 (July 1952).

The Perspirometer appears to offer an improvement in measuring fastness to perspiration of coloured textiles over British and Continental methods on the score of simplicity and speed. A modified model is described which costs approx. one-tenth the price of the A.A.T.C.C. instrument.

B. K.

Reduction of some Chlorinated Azobenzenes with Titanous Sulphate (IV p. 403).



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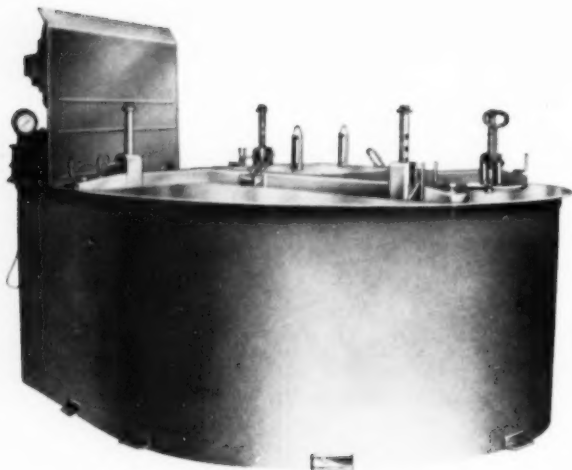
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FORTHCOMING MEETINGS OF THE SOCIETY—continued from page x

HUDDERSFIELD SECTION

All meetings held at Field's Cafe, Huddersfield, at 7.30 p.m., unless otherwise stated

- 1952
 Tuesday 21st Oct. G. G. Taylor, Esq. and J. C. Brown, Esq. (Clayton Aniline Co.). *Textile Microscopy*
 Friday 31st Oct. ANNUAL DINNER
 Monday 3rd Nov. Dr. C. S. Whewell. *The Future of Textile Finishing* (Joint with Huddersfield Textile Society)
 Tuesday 18th Nov. R. J. Hannay, Esq. (Brotherton & Co.). *The Use of Hydrolysable Esters in the Control of Dyebaths*
 Tuesday 2nd Dec. Sir Wallace Akers, F.R.S. *Chemistry and the Textile Industry* (Joint with R.I.C. Huddersfield Section)
 Tuesday 16th Dec. Professor J. B. Speakman. Title later
 1953
 Tuesday 20th Jan. G. H. Rostron, Esq., A.R.I.C. (J. Crosfield). *Textile Process Soaps*
 Tuesday 17th Feb. INTER-SECTIONAL DISCUSSION
 Tuesday 17th March Dr. H. A. Thomas (Courtaulds). *The Role of Dyeing, Printing, and Finishing in Fabric Development*
 Tuesday 7th April ANNUAL GENERAL MEETING followed by a lecture—Dr. J. F. Gaunt. *A Study of the Afterchrome Process of Wool Dyeing*

SCOTTISH SECTION

All meetings at St. Enoch Hotel, Glasgow, 7 p.m., unless otherwise stated

- 1952
 Tuesday 21st Oct. S. Burgess, Esq. *Economic, Technical and other Difficulties encountered in meeting Fastness Requirements*
 Thursday 30th Oct. Professor J. B. Speakman. Details later
 Tuesday 11th Nov. H. R. Hadfield, Esq. *A Review of the Dyeing of Terylene Polyester Fibre*
 Thursday 13th Nov. J. V. Summersgill, Esq. *Sequestering Agents and their Applications in Textile Processing*
 W. Beal, Esq. *An Investigation into the Dyeing Properties of Acid and Chrome Dye-stuffs and its Practical Significance*
 This meeting will be held in the Scottish Woollen Technical College, Galashiels, at 7.30 p.m.
 Tuesday 9th Dec. Ministry of Fuel and Power. *Steam Utilisation and Boiler Efficiency*
 1953
 Tuesday 13th Jan. J. C. Brown, Esq. *Microscopy for the Dyer and Finisher*

LEEDS JUNIOR BRANCH

All meetings to be held in the Colour Chemistry Lecture Theatre, The University, Leeds, 2 on Tuesdays at 4.0 p.m.

- 1952
 21st Oct. — — Sumner (I.C.I. Ltd.). *The Colour and Constitution of Anthraquinone Vat Dyes*
 25th Nov. C. O. Clark, Esq., F.T.I. *Historical Records*
 1953
 20th Jan. J. Boulton, Esq., M.Sc.(Tech), F.R.I.C., F.T.I. (Courtaulds Ltd.). Title to be announced.
 17th Feb. G. G. Taylor, Esq., B.Sc., A.Inst.P. and J. C. Brown, Esq. (The Clayton Aniline Co. Ltd.). *The Uses of Microscopy in Textile Dyeing and Finishing*
 3rd March D. Hanson, Esq., B.Sc., F.R.I.C. (J. Crowther & Sons Ltd.). *A Chemist in the Woollen Industry*

WEST RIDING SECTION

All meetings held at the Victoria Hotel, Bradford, at 7.15 p.m. unless otherwise stated

- 1952
 Thursday 30th Oct. A. Gaunt, Esq. and P. Robinson (T. F. Firth & Sons Ltd.). *Dyeing in the Carpet Trade*
 Thursday 13th Nov. H. C. Olpin, Esq. and A. J. Wesson, Esq. (British Celanese Ltd.). *Fugitive Tinting with particular reference to Celafibre*
 Tuesday 18th Nov. C. R. M. Oehlske, Esq. (Messrs Newcol). *The Use of Organic Colours in Modern Plastics*
 The University, Leeds
 Tuesday Nov. 25th Dr. R. L. Wormell (Courtaulds Ltd.). *Casine and Peanut Protein Fibres* (Joint lecture with the Yorkshire Section of the Textile Institute). Midland Hotel, Bradford
 Thursday 27th Nov. H. R. Hadfield, Esq. (Imperial Chemical Industries Ltd.). *A Review of the Dyeing of Terylene Polyester Fibre*
 Thursday 11th Dec. FILM EVENING. Ladies to be invited. (Joint meeting with Yorkshire Section Textile Institute)
 1953
 Friday 16th Jan. LADIES' EVENING
 Thursday 29th Jan. Lecture. Details later
 Thursday 12th Feb. D. P. Raper Esq. (Imperial Chemical Industries Ltd.). *Wool Dyeing Faults and their Correction*
 Monday 23rd Feb. N. R. Hjort, Esq. *Water Treatment*. (Joint lecture with the Halifax Textile Society). Alexandra Cafe, Halifax
 Thursday 26th Feb. J. V. Summersgill Esq. (Geigy Co. Ltd.). Title later
 Tuesday 3rd March Dr. H. Baines (Kodak Ltd.). *Colour Photography*. The University, Leeds
 Thursday 12th March Dr. J. F. Gaunt (Patons & Baldwins Ltd.). *A Study of the Afterchrome Process of Dyeing Wool*
 Thursday 26th March ANNUAL GENERAL MEETING

BRADFORD JUNIOR BRANCH

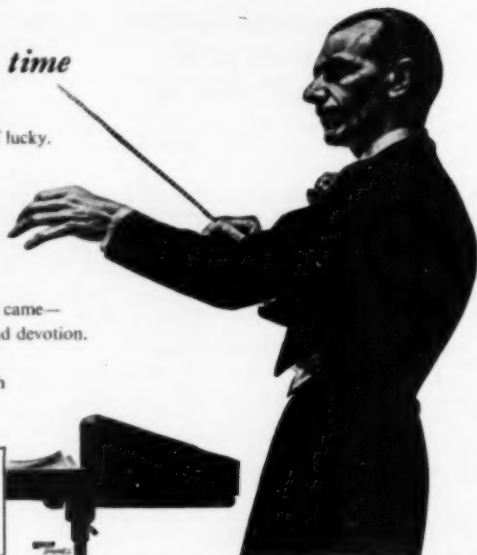
All meetings held in the Bradford Technical College at 7.15 p.m.

- 1952
 Wednesday 29th Oct. Visit to Messrs. Joseph Watson & Sons Ltd., Whitehall Soap Works, Leeds
 Friday 14th Nov. Dr. R. H. Peters (Imperial Chemical Industries Ltd.). *Recent Investigations into the Properties of Vat Dyes*
 Thursday 27th Nov. Visit to Salts (Saltaire) Ltd.
 Tuesday 9th Dec. FILM EVENING
 1953
 Monday 19th Jan. J. W. Fisher, Esq., B.Sc., F.R.I.C. (British Celanese Ltd.). *Synthetic Fibres*
 Wednesday 4th Feb. Visit to Messrs. Montague Burton Ltd., Hudson Road Mills, Leeds
 Wednesday 18th Feb. T. Green, Esq. (Clayton Aniline Co. Ltd.). *The Measurement of pH and its Importance in Relation to Textile Processing*
 Thursday 26th Feb. JUNIOR BRANCH DANCE at the Queens Hall
 Friday 13th March STUDENTS' ANNUAL COMPETITION EVENING
 Thursday March 26th FILM EVENING

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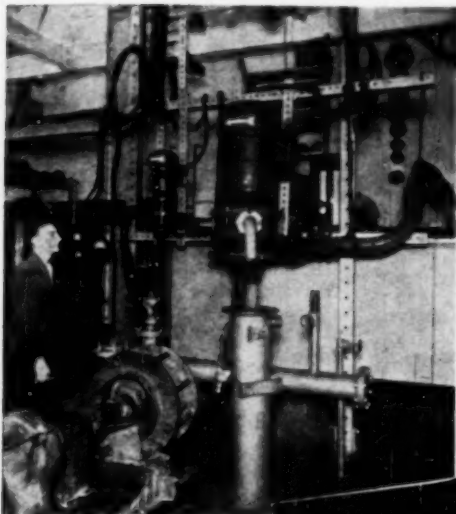
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